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USE OF FLAX FIBRE AS A REINFORCEMENT FOR CONCRETE

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Prepared for: Saskatchewan Ministry of Agriculture

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Summary

The vast majority of the flax straw produced in Saskatchewan is currently treated as a waste material and is burned or otherwise discarded. Previous research has demonstrated that the fibre within the straw can be used as a replacement for synthetic fibres to effectively reduce the cracking of fresh concrete (plastic shrinkage cracking), which can serve to improve the concrete's longevity. For this application, the fibres have served their purpose within the first day after the concrete has been placed. However, the unknown effect of the gradual deterioration of the fibre within the highly alkaline concrete environment has thus far prevented its acceptance for this application. The long-term goal of this research program was to develop a technically feasible flax fibre-reinforced concrete (FRC). Three specific objectives were targeted by this project: (i) to quantify the deterioration of flax fibre within a concrete environment, (ii) to quantify the effect that flax fibre degradation has on the long-term durability and mechanical properties of flax FRC, and (iii) to identify effective methods for improving the durability of flax fibre in concrete.

The first phase of tests focused on how the flax fibre itself, both in raw form and when subjected to four chemical treatments (mercerization, silane, acrylation, acetylation), was affected by exposure to a highly alkaline concrete-like environment. Samples of treated and untreated fibre were immersed in a synthetic concrete porewater solution with a pH of 13.5 at 38°C for time periods ranging from 0 to 112 days, with measurements of mass loss, colour changes, tensile properties, and water retention taken at regular intervals. The properties of the fibre were observed to deteriorate continuously over time as a result of the fibre bundles (technical fibres) breaking down into individual plant cells (elementary fibres) as the constituents binding them together were chemically dissolved. After 112 days of immersion, untreated fibres experienced a 23% mass loss, 86% loss of tensile strength, and 42% increase in water retention. None of the four treatments was effective at protecting the fibre from deterioration; treated fibres experienced losses of tensile strength ranging from 82 to 96% after 112 days of exposure.

The second phase of tests focused on the durability of concrete containing flax fibre in three forms (untreated, silane treated, Duralin treated) when subjected to 50 cycles of wetting and drying over 250 days, or 300 cycles of freezing and thawing over 90 days. This phase demonstrated that the breakdown of fibres observed in Phase 1 did not result in the degradation of the properties of the concrete over time. Changes to the compressive strength, dynamic modulus of elasticity, and flexural strength of concrete specimens containing untreated flax fibre did not differ significantly from those experienced by plain concrete specimens for any of the weathering scenarios tested. Furthermore, neither of the two fibre treatments had a significant influence on any of the investigated properties of the flax fibre-reinforced concrete specimens. Confirming previous research, the fibres did not impart significant levels of flexural toughness to the concrete, and neither weathering nor fibre treatments influenced this result significantly.

This study produced no evidence that the presence of flax fibres will negatively affect the properties of concrete over time, despite the fact that the fibres themselves may degrade. It can therefore be concluded that flax fibre is a technically feasible alternative to synthetic fibres for the control of plastic shrinkage cracking and that it can be used in untreated form for this application.

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1. Introduction

1.1. Background

Soon after concrete has been placed, when it is still in a fluid or 'plastic' state, it has a tendency to shrink as it cures, particularly when it is placed in hot, dry, and/or windy conditions. Because shrinkage is usually prevented by other structural elements or underlying support, the concrete is prone to cracking. The development of the so-called plastic shrinkage cracks can detrimentally affect the durability of concrete components because water can more easily penetrate into the concrete through the cracks, carrying with it aggressive substances such as deicing salts and reducing the concrete's resistance to damage caused by the expansion of freezing water. Concrete pavements, bridge decks, airport runways, and parking garage floors are particularly vulnerable to this type of cracking because of their large surface areas. In addition to proper finishing and curing practices, the most effective means of controlling plastic shrinkage cracking has been found to be the addition of relatively small amounts (less than 0.5% by volume) of short fibres, typically made of a polymer or glass (Folliard and Simpson 1998, Soroushian et al. 1995). The fibres provide some additional tensile capacity for the fresh concrete, thereby reducing the number of cracks that form, and also bridge the cracks that do form and reduce their widths.

While synthetic fibres are generally used for this application, several published studies have shown that fibres derived from natural sources can provide similar benefits. For example, Toledo Filho and Sanjuan (1999) found that the addition of 25 mm long sisal fibres at 0.2% by volume were as effective as polypropylene fibres in controlling plastic shrinkage cracking. Soroushian and Ravanbakshs (1998) reported that cellulose fibres at 0.06% by volume reduced plastic shrinkage crack area by 78 percent relative to unreinforced concrete. Previous research conducted at the University of Saskatchewan has demonstrated that fibres derived from flax straw are as effective at limiting the development of plastic shrinkage cracks as other commercially available fibres produced specifically for this purpose (Boghossian and Wegner 2008).

However, the durability of flax fibres in concrete is a concern that must be addressed before they can be seriously considered for this application. The durability-related issues arise due to the composition and structure of both the fibre and the cement paste of the concrete. The structure of the flax fibre has been described by several authors (Bledzki and Gassan 1999, Bos et al. 2002, Jähn et al. 2002, Pejic et al. 2008). As shown in Fig. 1, the stem of the flax plant is composed of fibres that are present at several different length scales. The elementary flax fibre is a single plant cell, whose structure and constituent materials are similar to those of other plants, although its dimensions and the relative proportions of constituent materials are different. The elementary flax fibre is 20 to 50 mm in length, with a polyhedral cross-section having a 'diameter' typically between 10 and 25 μm (Bos et al 2002). Its primary constituent materials are (Bledzki and Gassan 1999): cellulose (65-70% w/w), a highly crystalline linear glucose polymer with a degree of polymerization on the order of 8000; hemicellulose (16%), a mixture of different lower molecular weight branched polysaccharides; lignin (3%), a complex hydrocarbon polymer; and pectin (3%), consisting of polygalacturon acid.

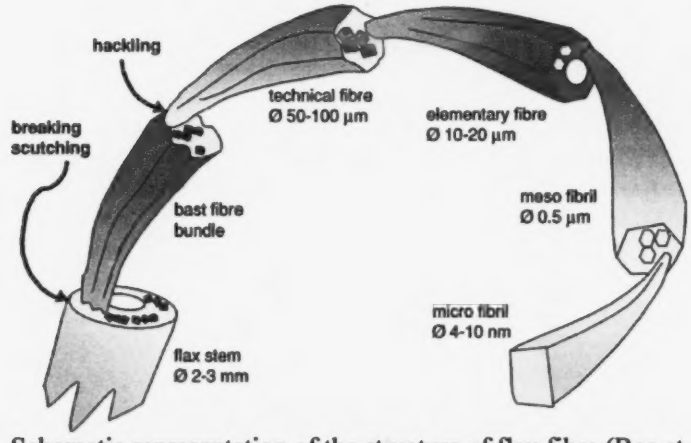


Figure 1. Schematic representation of the structure of flax fibre (Bos et al. 2002).

As seen schematically in Fig. 2, the elementary fibre is composed of several cell wall layers formed around an open channel (the lumen). The primary cell wall is a relatively thin layer (~0.2 µm) comprising significant amounts of pectin and lignin, in addition to hemicellulose and cellulose. The secondary cell wall makes up most of the fibre thickness and comprises a network of ultrafine cellulose micro-fibrils, packed together in fibrillar bundles (meso-fibrils), most of which are spirally oriented at an angle of approximately 10° around the axis of the fibre. Hemicellulose occupies the spaces between the cellulose fibrils, and serves to bind them together. Individual elementary fibres are glued together in bundles of 10 to 40 by the middle lamella, which consists mainly of pectin, hemicellulose, and lignin, to form the technical fibre, with diameters greater than 50 µm.

The cement paste in concrete is a very porous material. When partly or fully saturated, the porewater is highly alkaline, with a pH on the order of 13, due to dissolution of hydroxides (potassium, sodium, and calcium) from the cement paste. When natural fibre is exposed to a highly alkaline environment, its constituent materials are subject to degradation. Several mechanisms have been identified as being responsible for changes to fibre properties when embedded in a cement matrix (Bentur and Akers 1989, Gram 1988): (i) alkaline hydrolysis of the cellulose molecules, leading to a reduction in the degree of polymerization, and therefore a reduction in tensile strength, (ii) dissolution of the pectin, lignin and hemicellulose in the middle lamella, breaking the bond between individual elementary fibres,

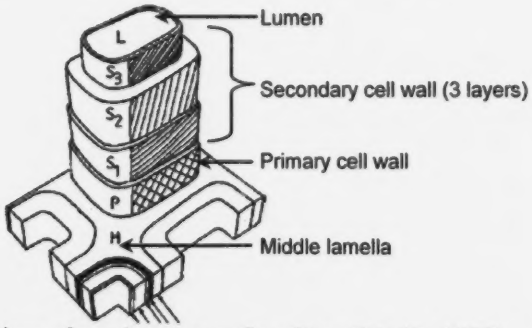


Figure 2. Schematic view of an elementary flax fibre (Fördös 1988).

and effectively disintegrating the technical fibres, and (iii) mineralization of the fibre as it absorbs calcium hydroxide from the porewater, causing the fibre to become stiff and brittle over time.

The extent of this degradation and its effect on the mechanical properties of flax fibre are not yet completely known. Since the degradation takes some time to occur, it will not be detrimental to the fibre's ability to control plastic shrinkage cracking, as this function occurs within the first several hours after casting. However, it is not known whether the presence of the fibre will negatively influence the properties of the concrete over the long term. It is also not known whether it is possible to protect the fibre against alkali attack by means of some sort of fibre treatment in order to minimize or eliminate the degradation.

A second benefit of the addition of small amounts of fibre to concrete is the associated increase in flexural toughness. This is a property that allows the concrete to sustain some load-carrying capacity after it has cracked and thereby to absorb energy as it deforms. Flax fibre in its raw state has been shown not to perform this function well, because the bond between the fibre and hardened cement paste is too strong and the fibre has a relatively low capacity to stretch and therefore sustain a tensile load across a widening crack (Wang and Wegner 2001). This is exacerbated over time as the bond strength increases with the absorption of the cement paste constituents (Bentur and Akers 1989). However, it is possible that treatments that protect the fibre from alkali attack could also serve to reduce the fibre-cement bond, thereby increasing the flexural toughness. The degradation of fibres over time may also weaken their bond to the surrounding cement paste, which would also have the effect of increasing the flexural toughness. This would be an added benefit that would open up additional applications for flax fibre-reinforced concrete and increase the demand for flax fibre in the construction industry.

1.2. Objectives

The long-term goal of this research program is to develop a technically feasible and durable fibre-reinforced concrete (FRC) that incorporates fibre derived from flax straw. This project specifically targeted the following objectives:

(i) To quantify the deterioration of flax fibre when exposed to concrete porewater;

(ii) To quantify the effect that flax fibre degradation has on the long-term durability and mechanical properties of flax FRC; and

(iii) To identify effective methods for improving the durability of flax fibre in concrete.

2. Experimental Methods

2.1. Overview

The research program was carried out in two phases. In the first phase, four chemical treatments were identified as candidates to protect the fibre from alkali attack. After application of these treatments, samples of untreated and treated fibre were immersed in a synthetic concrete porewater solution for up to 112 days. At regular intervals, samples of each type of fibre were removed from the solution and tested to quantify the level of degradation. Measurements included mass loss, colour changes, water retention and tension

properties (tensile strength and Young's modulus). In addition, scanning electron microscopy (SEM) was used to identify any changes in the appearance of the fibre surface that could be caused by degradation.

Phase 2 of the project focused on measuring the properties of concrete reinforced with untreated and treated fibre before and after specimens were subjected to two types of cyclic weathering scenarios: wetting and drying, and rapid freezing and thawing. Two different fibre treatments were evaluated in this phase. The degradation of mechanical properties was quantified using measurements of compressive strength and Young's modulus. In addition, flexural tests were used to measure the modulus of rupture and flexural toughness.

2.2. Phase 1 - Degradation of Flax Fibre in Synthetic Concrete Porewater Solution

2.2.1. Materials

The flax fibre used in this study was grown in Saskatchewan and was obtained from Biolin Research, Inc (Saskatoon, SK). The fibre used for the first phase of experiments had an initial shives content of 25% by mass, which was reduced to approximately 11% after manual separation and cleaning. The shives content was determined by completely removing the shives from a specified weight of flax fibre, and then weighing the fibre and shives to the nearest gram separately. Based upon observation of the size of individual fibres, the fibre used was a combination of technical fibre bundles and elementary fibres, which separated from the bundles during processing.

2.2.2. Fibre Treatments

All fibre was cleaned by washing in a 2% detergent solution (Liquid Tide®), followed by rinsing in distilled water, and then oven drying at 70°C for 24 hours. Untreated fibre samples were tested in this condition, without any further processing.

Four different chemical treatments were applied: mercerization, silane, acrylation, and acetylation. Mercerization, also known as alkaline treatment, refers to the process of subjecting the fibre to a concentrated basic aqueous solution. The treatment increases the surface roughness of the fibre and removes a certain amount of the lignin, wax and oils from the external surface of the cell wall. In addition, it removes some lignin and hemicellulose from within the cell walls, allowing improved orientation of the cellulose microfibrils. As a result, it has been found to improve the mechanical properties of flax fibre (Gassan and Bledzki 1999, Jähn et al. 2002, Li et al. 2007). All treated fibre (1 kg in total) was first subjected to mercerization by immersion in a 5% (by weight) sodium hydroxide solution for one hour, rinsing in distilled water, and oven drying at 70°C for 24 hours. One portion (250 g) of the fibre was tested in this condition without further treatment, while the remaining fibre was subjected to one of the three other treatments.

Silane (SiH_4) is a coupling agent normally used to improve the bond between glass fibres and a polymer matrix in glass fibre-reinforced composites. It has also been found to improve the surface properties of natural fibre (Li et al. 2007). For application of the silane treatment, 250 g of mercerized fibre was soaked in one litre of a 2.5% (by weight) triethoxysilane coupling agent solution (alcohol:water mixture at 60:40) for 30 minutes. The pH was continuously adjusted to between 3.5 and 4.0 using MetrePak pHydrion® buffers and

pH indicator strips. Finally, it was washed with distilled water and dried at 70°C for 24 hours.

Acrylation is the treatment of fibre with acrylic acid, which has been reported to provide a surface coating to natural fibres (Sreekala et al. 2000). This treatment was applied by immersing 250 g of the mercerized fibre in one litre of a 5% (by weight) acrylic acid solution for one hour at 50°C, followed by rinsing in distilled water and drying at 70°C for 24 hours.

Finally, acetylation is a well-known chemical modification process used to give natural fibre hydrophobic properties. It involves the reaction of acetic anhydride with the hydroxyl groups in the cell walls to cause plasticization (Rowell 2004, Bledzki and Gassan 1999, Li et al. 2007). For the current study, acetylation of the flax fibre was accomplished by immersing 250 g of fibre in one litre of a 5% (by weight) solution of acetic acid for one hour, followed by immersion in a 5% (by weight) acetic anhydride solution with two drop of sulphuric acid added as a catalyst. After 10 minutes, the fibre was rinsed in distilled water and dried at 70°C for 24 hours.

2.2.3. Sample Preparation and Conditioning

A synthetic concrete porewater solution was prepared by dissolving 0.32M potassium hydroxide, 0.17M sodium hydroxide and 0.006M calcium hydroxide in distilled water, producing a solution with a pH of 13.5, as measured using a Precise pH meter (PHS-3C). A total of 125 plastic jars were each filled with 200 mL of the solution, to which 5 g of fibre was added. A total of 125 samples were prepared, comprising 25 samples for each type of fibre treatment (five types, including the untreated fibre). After lids were applied, the jars were placed in a temperature controlled room and held at 38±2°C until they were to be tested. At conditioning times of 7, 14, 28, 56, and 112 days, five samples of each fibre type were removed for testing. A set of unconditioned samples of each type of fibre was also tested to provide initial baseline properties.

At the test intervals, each fibre sample was taken out of its plastic container and placed on a strainer lined with filter paper and set on a 2 L beaker. The excess solution was carefully squeezed from the fibre, taking care that no fibre was lost in the process. The pH of the remaining solution was measured using the same pH meter, and was found in all cases to have dropped by approximately 0.3 to 13.2. The wet fibre was placed on a filter paper and dried at 50±2°C for 24 hours in a low heat oven, which incorporated two dehumidifiers in series to circulate the air and maintain a low humidity.

2.2.4. Test Procedures

2.2.4.1. Mass Loss

Mass loss is a measure of the amount of the fibre constituents that are broken down and removed by the solution. The mass of each oven-dried sample was measured to the nearest 0.01 g, and the percentage mass loss relative to the initial 5.00 g mass was calculated as follows:

$$\Delta m = \frac{m_i - m_f}{m_i} \times 100 \qquad (1)$$

where m_i and m_f are the initial (5.00 g) and final masses of the sample, respectively.

A portion of the mass loss as calculated above could be the result of handling processes, rather than due to the effect of conditioning in the alkaline solution. To estimate this portion, four 2 g samples of unconditioned fibre from each of the five fibre treatment types were soaked in distilled water, squeezed and dried in the same manner as described in the previous section. The mass of each sample was measured before and after these handling processes were performed, and mass loss due to handling was calculated using Eq. 1.

2.2.4.2. Colour Changes

Changes in the colour of a fibre sample may reflect changes to its chemical makeup as a result of degradation. Increasing levels of degradation are therefore expected to produce more significant colour changes. The colour of each sample was measured four times in succession, using a HunterLab spectrocolorimeter (LABSCAN2, Hunter Associates Laboratory Inc., Reston, VA). The colour of 1.5 g of fibre, placed on a transparent dish, was measured after calibration of the instrument against both white and black ceramic plates. The average of the five replicate samples for each treatment at each interval was used. Unconditioned fibre of the corresponding type of treatment was used as the standard for comparison of fibre with the same treatment.

As shown in Fig. 3, the HunterLab spectrocolorimeter uses three parameters to indicate the colour of a sample. The *L* parameter indicates the lightness of the sample, with a value of 0 corresponding to black and a value of 100 corresponding to white. The parameter *a* describes the colour in terms of the relative intensity of green and red, with negative and positive values indicating green and red, respectively. Finally, the *b* parameter describes the relative amounts of blue and yellow, with negative and positive values indicating blue and yellow, respectively.

The differences in these three parameters were calculated as follows:

$$\begin{aligned} \Delta L &= L_f - L_i \\ \Delta a &= a_f - a_i \\ \Delta b &= b_f - b_i \end{aligned} \tag{2}$$

where the '*i*' subscript refers to values for the unconditioned fibre, and the '*f*' subscript refers to the conditioned fibre. The total colour change was then calculated as

$$\Delta C = \sqrt{\Delta L^2 + \Delta a^2 + \Delta b^2} \tag{3}$$

2.2.4.3. Tension Properties

The single fibre tension tests were used to provide a direct measure of changes in the mechanical properties of the fibre as a result of exposure to the porewater solution. From the dried samples, 20 individual strands of fibre, each greater than 72 mm long, were selected for each type of treatment at each exposure period. These were mounted in a paper frame by epoxy gluing the two ends to opposite sides of the frame, as shown in Fig. 4. In order to estimate the cross-sectional area of each fibre sample, the fibre was assumed to have a circular cross section, and the diameter of each strand was measured to the nearest 0.01 μm

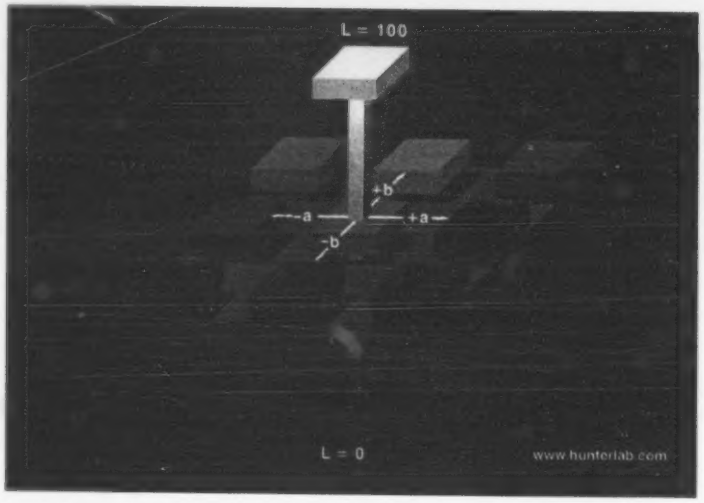


Figure 3. Colour chart illustrating the LAB colour system (Hunter Associates Laboratory, Inc. 2008)

at three locations along the length using a digital image obtained by an optical microscope (Nikon Optiphot) and digital camera (Q-imaging 10bit) at a magnification of 100X.

Tension tests were performed by clamping the two ends of a frame in a texture analyzer (Texture Technologies Corp., Scarsdale, NY), cutting the paper frame on each side of the fibre, and applying a tensile load to the fibre at a crosshead speed of 0.05 mm/s until it ruptured. A gauge length of 72 mm, equal to the distance between grips, was used for strain calculations, taking the crosshead displacement to be equal to the extension of the fibre during the test. The applied force was measured to the nearest 0.001 N. Tensile strength was obtained by dividing the maximum force by the minimum cross-sectional area measured for a given fibre, while Young's modulus was taken as the change in stress divided by the change in strain from the beginning to the end of the test. Some of the prepared fibre samples were discarded prior to testing due to their poor condition, and a maximum of one value from each set of replicate tests was removed if it was identified as an outlier using Grubbs' test (Barnett and Lewis 1994), leaving a minimum of 14 measurements from which to calculate average tensile strength and Young's modulus.

2.2.4.4. Water Retention

Water retention tests were performed to measure the amount of water the fibre absorbs

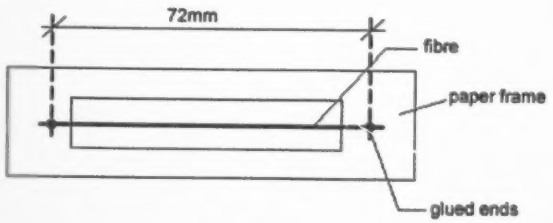


Figure 4. Schematic representation of a flax fibre glued to a paper frame for the tension tests.

relative to the dry mass of the fibre. A fibre treatment that reduces water retention is expected to provide the fibre with greater resistance to degradation, since degradation is the direct result of the chemical reaction between the highly alkaline aqueous solution and fibre constituents. In addition, higher levels of water retention could reduce the freeze-thaw resistance of concrete with embedded fibres.

Water retention was measured following ASTM Standard D2402-07 (Standard Test Method for Water Retention of Textile Fibers (Centrifuge Procedure)). From each dried 5 g sample, 2±0.001 g of fibre were picked randomly and immersed in distilled water for five minutes to fully saturate it. The fibre was then removed from the water and placed in a 60 mL syringe with the plunger removed. This, in turn, was placed inside a syringe tube, providing a place for the removed water to collect (see Fig. 5). The syringe and tube were then placed in a centrifuge, which was run for five minutes at an acceleration of 9800 m/s^2 to force water out of the syringe and into the tube. This process mechanically removed surface water from the fibre using a standardized force. The mass of the wet fibre (m_w) was then measured to the nearest 0.001 g, the fibre was dried at 50±2°C in a low-heat oven for 24 hours, and the dry mass (m_d) was measured. Water retention was calculated as a percentage of the dry mass:

$$wr = \frac{m_w - m_d}{m_d} \times 100 \tag{4}$$

2.2.4.5. Scanning Electron Microscopy

Scanning electron microscopy (SEM) was used to identify microstructural changes that may have occurred on the fibre surfaces as a result of exposure to the porewater solution. Small samples of fibre with each of the different fibre treatment types and at each time interval were gold coated and then observed using a JEOL 840A SEM (JEOL Ltd., Tokyo, Japan) operating at 15 kV and located in the Department of Geological Sciences at the University of Saskatchewan.

2.2.5. Changes from Original Work Plan

The following relatively minor changes were made to the original work plan for this phase:

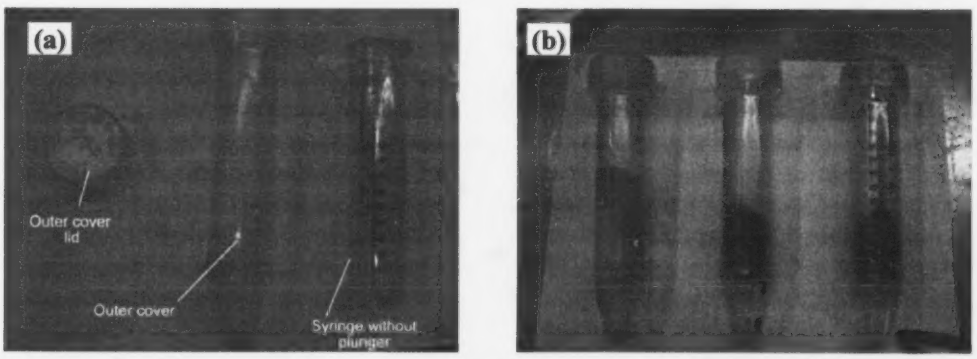


Figure 5. Containers used for the water retention tests: (a) the component pieces, and (b) the assembled apparatus.

- A synthetic porewater solution was used rather than a solution produced by dissolving crushed hardened cement paste in water, as was originally proposed. This approach is commonly found in the literature, and was done to ensure a more consistent composition of the solution.
- The original work plan proposed a set of pre-screening tests to identify potential fibre treatments. This set of tests was not performed. Only four potential fibre treatments were identified, and in order not to delay the start of detailed testing, all four were used for the detailed tests. Based on the results obtained at the earliest tests intervals, preferred treatments could not have been identified using the proposed approach.
- Sample sizes that differed from those proposed were used, based on available equipment and further study.
- Measurement of colour changes in the fibre samples was not part of the original work plan. This could be carried out very quickly and provided an additional indicator of deterioration.

2.3. Phase 2: Durability Tests on Flax Fibre Reinforced Concrete

2.3.1. Overview of Phase 2

The influence of two different weathering scenarios—wet-dry cycling and freeze-thaw cycling—on the durability of flax fibre-reinforced concrete (FFRC) specimens was evaluated in this phase of the investigation. These weathering scenarios were intended to simulate long-term harsh exposure conditions that could be experienced by concrete elements, and were therefore expected to accelerate the degradation processes. The wet-dry cycling was performed in the Structures Laboratory at the University of Saskatchewan, while the freeze-thaw cycling was conducted in the research laboratory of Lafarge Canada Inc. in Richmond, British Columbia, where the necessary equipment was located.

Four different materials were tested, including plain concrete (without fibres), and specimens with three different forms of flax fibre: untreated, chemically treated with silane, and heat treated following a specified procedure known as the Duralin process. For each material and weathering scenario, the mechanical properties were measured after a standard 28 day moist curing period, after being subjected to the specified weathering scenario, and at the same age as the weathered samples but without having been subjected to the weathering. To evaluate the performance of each material, tests were conducted to measure compressive strength, dynamic modulus of elasticity, flexural strength, and flexural toughness. In addition, scanning electron microscopy (SEM) was used to examine the failure surfaces of flexural test specimens, and energy dispersive X-Ray spectroscopy was used to identify any changes to the chemical composition of the embedded flax fibres.

2.3.2. Materials and Specimen Preparation

2.3.2.1. Fibres and Treatments

The same flax fibre was used for this phase of the tests as for Phase 1. However, for Phase 2 tests, the fibre was processed mechanically using recently acquired equipment located in the Department of Agricultural and Bioresources Engineering Laboratory at the University of Saskatchewan. Three machines were used progressively clean the fibre: a

picker (model PKR, Belfast Mini Mills, Prince Edward Island), small fibre separator (model SFS, Belfast Mini Mills, P.E.I.), and large carder (model LCR, Belfast Mini Mills, P.E.I). These processing steps removed virtually all shives from the fibre. After cleaning, the fibres were cut to 38 mm lengths using a slicing machine (model CTR, Belfast Mini Mills, P.E.I.) located in the same laboratory. After these processes, approximately 2 kg of cleaned and chopped flax fibre was available for use.

As mentioned in the previous section, in addition to the raw flax fibre, two different fibre treatments were evaluated: chemical treatment with silane, and heat treatment following a procedure known as the Duralin process. The silane treatment was applied in a manner identical to that described in Section 2.2.2. The Duralin process, described in Stamboulis et al. (2001), was applied by first placing the cleaned and chopped fibre in an autoclave (scientific pre-vacuum sterilizer, model SV-120, Steris Corporation, Mentor, OH) and subjecting it to steam at a temperature of 160°C for 30 minutes. The fibre was then dried in an oven (Iso Temp Oven model 630G, Fisher Scientific, Hampton, NH) for two hours at 150°C. The treatment is normally applied to flax straw, from which the technical fibre is subsequently removed. However, for this study, it was applied directly to the fibre. According to Stamboulis et al. (2001), the hemi-cellulose and lignin in the cell walls are depolymerised into lower molecular weight polymers during the first stage, and combine to form a water resistant resin during the second stage.

Prior to being mixed into the concrete, the size range of fibres observed suggested that a mix of technical and elementary fibres were present. Figure 6 is an SEM micrograph of an untreated flax fibre, showing a number of elementary fibres on the right, separating from a technical fibre on the left.

2.3.2.2. Concrete Materials

Concrete with a minimum 28-day compressive strength of 32 MPa was targeted for this study, in order to meet the requirements for exposure class C-2, as specified in CSA Standard A23.1-00 (Concrete Materials and Methods of Concrete Construction). This type of concrete is suitable for use in slabs or pavements, which can be susceptible to plastic shrinkage cracking. The mix proportions used are shown in Table 1.

Figure 6. Scanning electron micrograph of an untreated technical fibre separating into its component elementary fibres.

Table 1. Concrete mix proportions.

Component	Proportion
Type 10 (GU) portland cement	535 kg/m^3
Tap water	215 kg/m^3
Coarse aggregate (14 mm max)	825 kg/m^3
Fine aggregate	825 kg/m^3
High range water reducer	325 – 1250 mL/100 kg cement
Air entrainment	16 – 260 mL/100 kg cement
Flax fibre (38 mm long)	0.3% by volume

Coarse aggregate with a maximum size of 14 mm was used. Because specimens for the two different weathering scenarios were produced in different laboratories, the aggregates used for the two sets of tests were not the same. However, the tests were not designed to make comparisons between the two sets, so this was not considered an important factor. The coarse aggregate used to make specimens for the freeze-thaw weathering study did not meet the gradation requirements of CSA Standard A23.1-00, as approximately 25% less aggregate than required between 4.75 and 12.75 mm was present; however, this was not considered an important factor since the material was consistent for all specimens. MB AE 90 air-entrainment admixture and Glenium 7101 high range water reducer (superplasticizer) were used.

2.3.2.3. Specimen Preparation

Concrete for the wet-dry weathering study was mixed in the Structures Laboratory in the College of Engineering at the University of Saskatchewan, while concrete for the freeze-thaw weathering study was mixed at Lafarge's research laboratory in Richmond, BC. In both locations, motorized mixers with a capacity of 0.15 m^3 were used. The mixing procedure involved the following steps: (1) all aggregate was added to the mixer, (2) the mixer was run for 30 seconds, (3) half the batch water and all the air-entrainment admixture were added while the mixer was running, (4) the mixer was run for one minute and then stopped, (5) all the cement was added, mixing was resumed and continued for one minute, (6) the remaining water and admixtures were added and mixing continued for three minutes, (7) mixing was stopped for three minutes, (8) mixing was resumed, fibres were added (when required), and mixing was continued for at least two minutes, (9) if fibres were balling up, the superplasticizer was added and the concrete was mixed for at least ten more minutes.

When mixing was completed, the fresh concrete was tested for slump and air content, and then cast into forms. The general procedures for forming and curing concrete followed ASTM Standard C192 (Making and Curing Concrete Test Specimens in the Laboratory). Specimens for each of the two weathering scenarios were cast in four batches, one for each of the four materials tested. For the wet-dry study, the four batches were cast on four consecutive days; each batch produced 20 beams of dimensions 75 x 100 x 400 mm for flexural tests, and 20 100 mm diameter x 200 mm cylinders for compression tests, both cast in PVC moulds. For the freeze-thaw study, 12 beams and four cylinders were cast in each batch using steel and PVC moulds, respectively. The four batches were cast over two days. Slump and air content for each batch are listed in Table 2, showing that air content was relatively consistent among the different batches for the wet-dry specimens. However, the air content for the freeze-thaw specimens varied considerably, with untreated fibre specimens

having a relatively low air content and silane treated fibre specimens having a relatively high air content.

Table 2. Fresh concrete properties.

Batch	Wet-dry specimens		Freeze-thaw specimens	
	Slump (mm)	Air content (%)	Slump (mm)	Air content (%)
Plain	120	9.1	110	7.2
Untreated fibre	105	9.9	150	4.5
Duralin	95	9.7	55	7.8
Silane	90	8.4	110	12

After casting, the concrete specimens were cured in a humid environment for 24 hours, then removed from their forms and placed in a bath of water saturated with calcium hydroxide (3.0 g/L) at a temperature of 25 ± 3°C. After another 27 days, specimens were removed from the curing bath and were ready for testing.

2.3.3. Weathering Procedures

2.3.3.1. Wet-Dry Cycling

Specimens for the wet-dry cycling study were divided into five groups, each comprising 16 beams (four of each material) and 16 cylinders (four of each material). The first group was tested immediately after the 28 day curing period to provide a baseline of properties to which subsequent measurements could be compared. The second and third groups were tested after 50 and 25 wetting/drying cycles, respectively, while the fourth and fifth groups were set aside in the laboratory (22 to 27°C, 50% relative humidity) and tested together with the second and third groups, respectively, to act as control specimens for comparison. For specimens in the second and third groups, the dynamic modulus of elasticity was measured after every wet-dry cycle, as described below.

Wetting and drying was carried out in the laboratory facilities of the College of Engineering at the University of Saskatchewan. For the wetting portion of each cycle, specimens were immersed in a tap water bath at 25°C in the humidity chamber of the Concrete Materials Laboratory. Drying was performed at 100°C in a 28 cubic foot oven (SHEL LAB High Performance Oven, model 1685, Sheldon Manufacturing Inc., Cornelius, OR) located in the Geotechnical Laboratory. For the second group of specimens, subjected to 50 wet/dry cycles, one complete cycle took four days. The first nine cycles consisted of three days of wetting and one day of drying, while the remaining 41 cycles consisted of two days of wetting and two days of drying. This change was implemented after observing the changes in mass during each cycle, in order to provide time for more complete drying to occur. The third group of specimens was subjected to 25 cycles, each lasting for eight days. The first five cycles consisted of seven days of wetting and one day of drying, while the remaining 20 cycles consisted of six days of wetting and two days of drying. Due to a short delay in beginning the cycling and a 16 day delay in the middle of the cycling, specimens from both groups were tested at an age of approximately 250 days.

2.3.3.2. Freeze-Thaw Cycling

Specimens for the freeze-thaw study were divided into three groups. The first group comprised 16 beam specimens (four of each material) and 16 cylinders (four of each material). After casting and moist curing for 28 days in Lafarge's research laboratory in Richmond, BC, these specimens were moisture sealed in containers and shipped to the University of Saskatchewan for testing to provide a baseline for properties to which subsequent measurements could be compared. Due to the time required for shipping, these specimens were tested at 30 or 31 days of age. The second and third groups each comprised 16 beam specimens, four of each material. Specimens in the second group were subjected to 300 cycles of rapid freezing and thawing, while those in the third group were set aside in the laboratory (22 to 27°C, 50% relative humidity) for testing together with those in the second group to act as control specimens for comparison. These latter two groups of specimens were sealed and shipped to the University of Saskatchewan for testing after the 300 cycles of rapid freezing and thawing were completed. They were tested at an age of approximately 90 days.

The freeze-thaw cycling was performed following procedure A of ASTM Standard C666-03 (Standard Test Method for Resistance of Concrete to Rapid Freezing and Thawing). This procedure consisted of immersing each beam specimen in a container of water, slightly larger than the 75 x 100 x 400 mm specimen. All 16 specimens were then placed in a rapid freeze-thaw cabinet (model H-3185, Humboldt Manufacturing Co., Schiller Park, IL), together with two dummy specimens, one of which was used to monitor the internal temperature of specimens and control the cycling. The cabinet automatically cycled the specimens from 4°C to -18°C then back to 4°C, with each cycle lasting between three and six hours. At intervals ranging from 24 to 41, specimens were removed, containers were rinsed and filled with fresh water, and specimens were returned to the cabinet for further cycling.

2.3.4. Test Procedures

2.3.4.1. Compression Tests

Compression tests were performed on 100 mm diameter x 200 mm long cylinders according to ASTM Standard C39 (Compressive Strength of Cylindrical Concrete Specimens). Tests were performed at a loading rate of 0.2 kN/s in an Instron 600DX Universal Testing Machine located in the Materials Laboratory in the College of Engineering. A total of 80 cylinders from the wet-dry study and 16 cylinders from the freeze-thaw study were tested.

In addition to the cylinder tests, compression tests were also performed on 75 x 100 x 150 mm prisms, which were cut from the ends of the beam specimens after they had been tested in flexure. These non-standard tests were performed because the freeze-thaw cabinet could not accommodate the standard cylinders, and a measure of the changes to compressive strength of specimens subjected to freezing and thawing was desired. Two specimens from every beam (one from either end) were tested, making a total of 160 from the wet-dry study and 96 from the freeze-thaw study. These specimens were tested under the same conditions as the cylinders, with the compressive force applied in the direction of the long dimension.

2.3.4.2. Dynamic Modulus of Elasticity

The dynamic modulus of elasticity, E, was measured for all beam specimens before they were tested in flexure. In addition, E of each beam was also measured after every wet-dry cycle. The principle of this test is that the natural frequencies of a beam are related to the stiffness or modulus of elasticity of the specimen. The test procedure therefore consists of measuring the fundamental natural frequency of a particular mode of vibration, and using this to estimate the modulus of elasticity.

Tests were performed according to ASTM Standard C215-08 (Standard Test Method for Fundamental Transverse, Longitudinal, and Torsional Resonant Frequencies of Concrete Specimens) using the setup for measuring the transverse natural frequency. For this test, the beam was laid flatwise on two symmetrically located rubber supports separated by a 221 mm span. An accelerometer (EpiSensor FBA ES-U, Kinemetrics Inc., Pasadena, CA) was mounted on the top surface at the end of the beam, while a ball-peen hammer was used to apply an impact force at mid-span. The acceleration response was measured in the vertical direction and recorded at a sampling rate of 20,000 samples per second for two seconds using LabView software installed on a laptop computer. A Fast Fourier Transform (FFT) was performed on the acceleration time history to identify the fundamental natural frequency. Five repeated tests were performed, and the average natural frequency from the five tests was calculated. The relationship between the dynamic E and natural frequency f is as follows:

$$E = CMf^2 \tag{5}$$

where M is the mass of the specimen in kg, f is the average natural frequency in Hz, and C is a function of the geometry of the beam and took on a value of approximately 1050 /m for the beams used in this study, depending upon the dimensions of individual specimens. Equation 5 shows that the dynamic modulus of elasticity varies with the square of the natural frequency.

2.3.4.3. Flexural Tests

Flexural tests were performed according to ASTM Standard C1609-10 (Standard Test Method for Flexural Performance of Fiber-Reinforced Concrete (Using Beam with Third-Point Loading)) to measure the flexural strength (modulus of rupture) and flexural toughness of FFRC specimens. Two deviations from the standard were made. First, the standard specifies specimens with a square cross-section; beams used for the current study were 75 mm wide and 100 mm high, which corresponded to the standard size used for freeze-thaw tests. Second, the standard recommends that the minimum width of the beam be three times the length of the longest fibre. In the present case, the 75 mm width was only approximately twice the length of the fibres.

A schematic of the flexural test set-up is shown in Fig. 7. Tests were performed in third-point bending with a supporting span of 300 mm. As shown, the longer cross-sectional dimension of the beams (100 mm) was oriented vertically. A hydraulic universal testing machine (Satec Systems 60HVL) located in the Materials Laboratory in the College of Engineering was used to perform the tests, which were conducted under displacement control at a rate of 0.1 mm/min. In order to eliminate extraneous deflections due to seating and twisting of the specimen and elastic displacement of the load frame and supports, two linear variable differential transformers (LVDTs), one on either side of the specimen, were

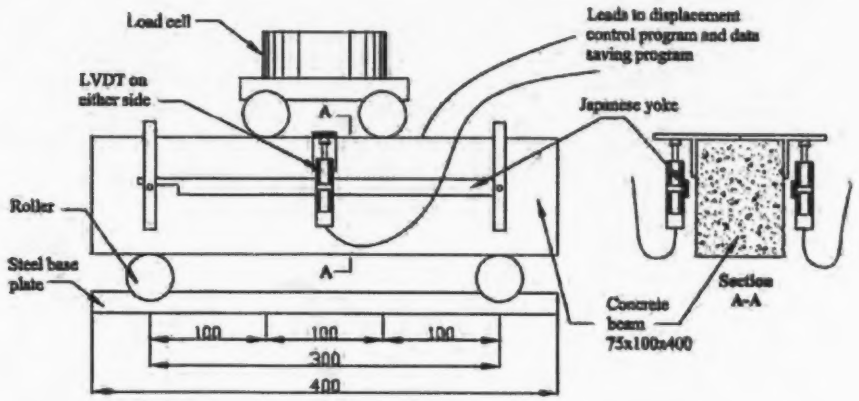


Figure 7. Schematic of the flexural test set-up (dimensions in mm).

mounted on a yoke that was supported on the top surface of the specimen immediately above supports. The LVDTs measured the relative displacement between this yoke and a saddle that was mounted on the specimen at mid-span. One of the LVDTs was used as the feedback control signal in order to maintain a constant displacement rate during the test. This type of displacement control, excluding any extraneous sources of displacement, is particularly important for capturing the response of the beam as cracking occurs.

Load and mid-span deflection were recorded at a sampling rate of 10 samples per second using LabView software implemented on a laptop computer that was separate from the one used for controlling the displacement rate. From the two deflection signals, the average mid-span deflection was calculated. The maximum recorded load was used to calculate the modulus of rupture (*MOR*) (also known as flexural strength) using the standard flexure formula, which simplified to the following equation for the test setup used:

$$MOR = 0.4P \tag{6}$$

where *MOR* is in MPa for a load *P* in kN.

As a measure of flexural toughness, two areas under the load-deflection curve were calculated, one corresponding to the peak load, typically accompanied by the appearance of the first crack (the pre-cracking toughness), and a second corresponding to the breaking load (the post-cracking toughness). While the pre-cracking toughness is a measure of a combination of the energy stored in the specimen and the smaller amount of energy dissipated prior to cracking, the post-cracking toughness is a measure of the energy dissipated as the crack widens prior to complete fracture. These calculations deviate from the provisions of ASTM Standard 1609-10, which specifies three different measures of the contribution of fibres to the post-peak response of the beams. However, the specified measures require the response at specimen deflections up to 2 mm. Unfortunately, the contribution of the flax fibres to the flexural response was relatively small, with deflections not reaching 0.1 mm for any of the beams tested.

2.3.4.4. Scanning Electron Microscopy

The failure surfaces of representative FFRC beams were observed in a scanning electron microscope (SEM) (JEOL 840A), located in the Department of Geology at the University of Saskatchewan, in order to better understand the fibre failure mechanisms. Samples with a 20 x 20 mm area were taken from a bottom corner of the failure surface of beams tested in flexure. A total of 12 samples were observed from the wet-dry cycling study, one sample for each of the three fibre types (untreated, Duralin, and silane treated) and four weathering scenarios (28 day curing, 50 four day cycles, 25 eight day cycles, ambient laboratory conditions). For the freeze-thaw study, a total of nine samples were studied, one for each of the three fibre types and three weathering scenarios. The samples were prepared by first removing moisture in a vacuum chamber and subsequently gold-coating them for electrical conductivity.

2.3.4.5. Energy Dispersive X-Ray Spectroscopy

In order to determine whether some of the cement constituents had migrated into the flax fibre, the chemical compositions of fibre samples were measured by means of energy dispersive X-Ray spectroscopy (EDS). These tests were conducted using a JEOL Superprobe electron microprobe analyzer located in the Department of Geology. Nine 20 x 20 mm samples were taken from the failure surfaces of beams from the wet-dry cycling study, one for each of the three fibre types and weathering scenarios (not including 28 day curing). In addition, three individual fibre samples, one for each treatment type, were analyzed. These samples were desiccated in a vacuum chamber and coated with carbon prior to testing.

2.3.5. Changes from Original Work Plan

Only relatively minor changes were made to the original work plan for this phase:

- For the freeze-thaw cycling study, non-standard block specimens were used to measure compressive strengths, since the freeze-thaw chamber could not accommodate standard cylinders.
- The dynamic modulus of elasticity was not measured correctly at regular intervals during the freeze-thaw cycling due to irregularities in the test procedure followed by staff at the Lafarge laboratory.

3. Results

3.1. Phase 1 - Degradation of Flax Fibre in Synthetic Concrete Porewater Solution

3.1.1. Mass Loss

Figure 8 shows the percentage mass loss experienced as a function of exposure time for fibres with all treatment types. Tabulated values are provided in Table 3. The values given for 0 days correspond to the mass loss resulting from handling processes without any exposure to the synthetic porewater solution. For the purpose of clarity, error bars representing the variability of measurements have been omitted from Fig. 8; Table 3 shows that coefficients of variation were consistently less than 1.5%, indicating that very little variability was experienced for mass loss measurements.

Table 3. Mass loss as a percentage of initial mass for all fibre treatments at all test intervals.

Treatment	Exposure Time (days)					
	0*	7	14	28	56	112
Untreated	6.98	9.12 (*0.7*)†	11.20 (*0.5*)‡	13.60 (*1.5*)	17.72 (*1.5*)	23.28 (*1.2*)
Mercerization	3.28	3.40 (*0.6*)	5.28 (*0.5*)	7.80 (*0.9*)	12.16 (*0.6*)	17.32 (*0.7*)
Silane	3.65	2.44 (*0.3*)	4.68 (*0.3*)	7.68 (*0.6*)	13.30 (*0.7*)‡	18.60 (*0.9*)
Acrylation	3.60	2.00 (*0.4*)	2.40 (*0.7*)	7.40 (*0.3*)	11.36 (*0.5*)	17.60 (*0.5*)
Acetylation	3.56	2.60 (*0.3*)	3.04 (*0.3*)	7.32 (*0.2*)	10.88 (*0.7*)	17.28 (*0.7*)

All values were determined from five replicate measurements, except when an outlier was removed.
* The value reported at 0 days corresponds to the mass loss due to handling processes.
† The italicized values shown in parentheses are the coefficients of variation in percent.
‡ Indicates that an outlier was removed.

It is clear that all four treated fibres experienced mass loss at a similar, almost linear, rate with time. The untreated fibre experienced a more rapid mass loss initially (approximately 9% within the first seven days), followed by mass loss at a similar rate to the treated fibres. The rapid initial mass loss corresponds to higher handling losses (7% compared to 2.5 to 3.5% for the treated fibres), and likely also, in part, to the early dissolution of the lignin, pectin, and waxes from the surfaces of the untreated fibres. On the other hand, the treated fibre would have had a certain amount of these constituents removed during the mercerization process, which was applied to all treated fibres. Once these were removed, a similar rate of mass loss was observed for all untreated and treated fibres. None of the treatments appears to effectively mitigate mass loss. Moreover, there is no sign of the rate of mass loss decreasing significantly, even after 112 days of exposure.

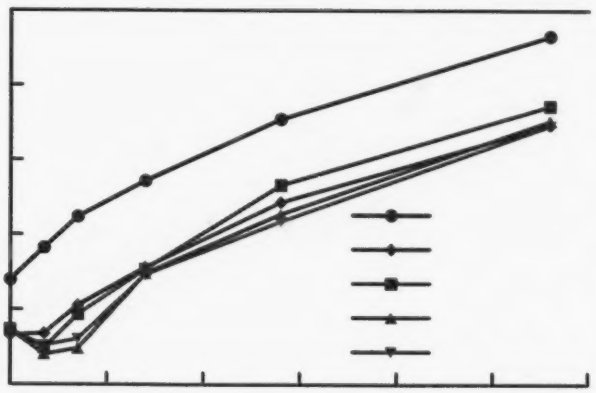


Figure 8. Variation of mass loss with exposure time for all fibre treatments.

3.1.2. Colour Changes

Measured colour changes are listed in Table 4 and plotted in Fig. 9. The relatively high coefficients of variation indicate that colour changes were subject to substantial variability. Nevertheless, fibres with all treatment types experienced statistically significant changes in colour. However, at 112 days of exposure, there was no significant difference between the colour changes experienced by any of the treated fibres and that of the untreated fibre. Figure 10, which plots the change in the lightness value, ΔL, shows that all fibre types became significantly lighter over time. Tabulated lightness values, L, are available in Appendix A.

Table 4. Total colour change, ΔC, for all fibre treatments at all test intervals.

Treatment	Exposure Time (days)					
	0	7	14	28	56	112
Untreated	0.0	0.6 (*128*)†	1.9 (*128*)	2.8 (*127*)	4.9 (*47*)	12.0 (*31*)
Mercerization	0.0	3.7 (*88*)	3.5 (*45*)	9.7 (*34*)	11.6 (*27*)	14.8 (*25*)
Silane	0.0	1.3 (*82*)	3.1 (*26*)	7.2 (*47*)	9.3 (*19*)	12.0 (*20*)
Acrylation	0.0	5.7 (*105*)	4.3 (*63*)	2.1 (*97*)	8.8 (*32*)	12.4 (*13*)
Acetylation	0.0	5.1 (*43*)	2.6 (*36*)	3.2 (*137*)	10.5 (*32*)	10.9 (*22*)

† The italicized values shown in parentheses are the coefficients of variation in percent.

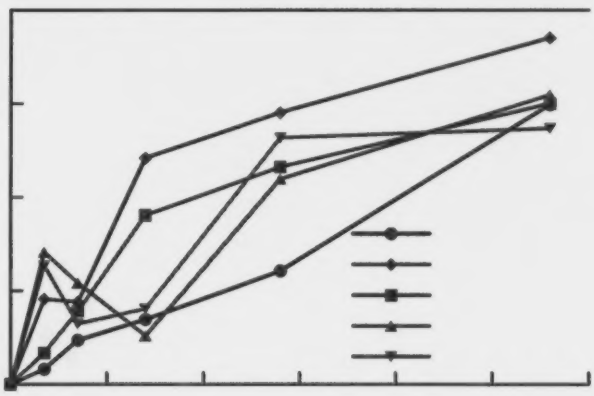


Figure 9. Colour changes with exposure time for all fibre treatments.

3.1.3. Tension Properties

Table 5 shows the measured tensile strengths for all fibres tested, while Fig. 11 shows the same results in graphical form to better visualize the trends. The coefficients of variation shown in Table 5, the majority of which lie between 60 and 90%, indicate a high level of variability in tensile strengths measured for a given set of replicate tests. In fact, as Table 5 shows, a relatively large number of outliers were identified. Given the number of outliers observed, these may not, in fact, be outliers in the normal sense of anomalous data, but may instead be more indicative of the high level of scatter in the results. To maintain clarity, error bars to indicate the variability are omitted from the figure.

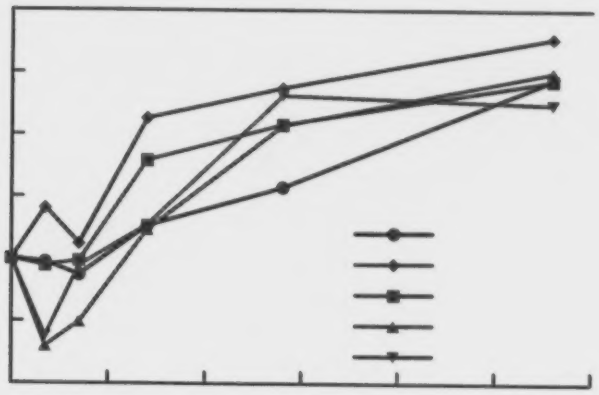


Figure 10. Changes to the lightness value, L, with exposure time for all fibre treatments.

Table 5. Tensile strength (MPa) for all fibre treatments at all test intervals, including data from all fibres tested.

Treatment	Exposure Time (days)					
	0	7	14	28	56	112
Untreated	119 (*79*)†‡	135 (*103*)	51 (*64*)‡	61 (*55*)‡	64 (*89*)	17 (*65*)‡
Mercerization	177 (*62*)	75 (*89*)‡	64 (*63*)	37 (*88*)	25 (*84*)‡	6.9 (*62*)‡
Silane	56 (*82*)‡	58 (*55*)‡	67 (*66*)	45 (*81*)‡	34 (*83*)	10 (*78*)‡
Acrylation	88 (*67*)‡	78 (*76*)	82 (*71*)	30 (*56*)‡	81 (*86*)	12 (*80*)
Acetylation	114 (*73*)	72 (*78*)‡	52 (*32*)‡	57 (*70*)	87 (*68*)	7.7 (*64*)‡

All values were determined from a minimum of 14 replicate measurements.
† The italicized values shown in parentheses are the coefficients of variation in percent.
‡ Indicates that an outlier was removed.

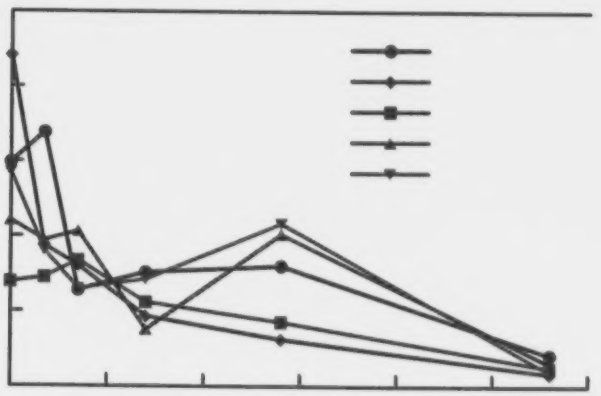


Figure 11. Change in tensile strength with exposure time for all fibre treatments.

Despite the scatter in the results and inconsistencies in the trends between different test intervals, there was a clear and significant loss of tensile strength experienced by all fibre types with increasing exposure time. Over 112 days, the untreated fibres lost 86% of their initial strength, while treated fibres lost between 82 and 96% of their initial strengths. Notwithstanding the high levels of variability, these reductions are significant at the 95% level of confidence. Moreover, with the exception of the acrylated fibres, the tensile strengths of all treated fibres were significantly smaller than that of the untreated fibre at 112 days at the 95% level of confidence. This indicates, firstly, that none of the treatments was effective at mitigating the loss of tensile strength, and, secondly, that the treatments apparently exacerbated the degradation. In addition, none of the fibre treatments, except perhaps for acrylation, appears to be superior to the others at 112 days of exposure.

A definite explanation for the apparent increase in tensile strength for the acrylated and acetylated fibre at 56 days has not been identified. It is possible that the selection of samples for testing was influenced by the deteriorating state of the fibre as time progressed, with the more durable and stronger fibres inadvertently selected for testing. As discussed below, over time, it became increasingly difficult to obtain samples of the required length as the fibres deteriorated with increasing porewater exposure.

Table 6 and Fig. 12 show the measured elastic moduli for all fibres tested. The trends are very similar to those observed for tensile strength. In this case, coefficients of variation generally lay between 45 and 90%, again indicating a high level of variability. However, the elastic modulus of all fibres decreased significantly over the 112 day testing period. In addition, at 112 days, the elastic modulus of the untreated fibre remained significantly greater than those of all of the treated fibres at the 95% level of significance, while none of the treatments resulted in a final elastic modulus that was significantly superior to that of the others.

Table 6. Elastic modulus (GPa) for all fibre treatments at all test intervals, including data from all fibres tested.

Treatment	Exposure Time (days)					
	0	7	14	28	56	112
Untreated	6.17 (*54*)†‡	7.20 (*71*)	4.02 (*88*)‡	4.05 (*45*)	4.90 (*45*)	1.60 (*70*)‡
Mercerization	9.80 (*65*)	5.45 (*68*)	3.44 (*67*)‡	0.94 (*61*)‡	1.87 (*61*)‡	0.49 (*88*)‡
Silane	3.47 (*95*)	4.32 (*41*)‡	4.45 (*55*)	2.78 (*79*)	2.30 (*62*)	0.72 (*88*)‡
Acrylation	5.52 (*58*)‡	4.64 (*39*)	3.61 (*64*)	2.06 (*61*)‡	6.28 (*71*)‡	0.47 (*72*)
Acetylation	6.83 (*68*)‡	4.60 (*54*)	3.10 (*55*)‡	2.35 (*44*)‡	4.59 (*55*)‡	0.42 (*60*)‡

All values were determined from a minimum of 14 replicate measurements.
† The italicized values shown in parentheses are the coefficients of variation in percent.
‡ Indicates that an outlier was removed.

Initially, the fibres failed in a brittle manner, exhibiting small elongations and sudden failures. However, they became less and less brittle after increased exposure to porewater. At 112 days, the majority of the failures were not brittle and more elongation was observed. In addition, it was increasingly difficult to obtain samples of the required 72 mm length after they had been exposed to the porewater solution. After exposure, the smaller elementary fibres that make up the technical fibre bundles did not hold together very well and came apart very easily when handled. This was most pronounced for fibres with the silane treatment. It appears that the middle lamella that bonds the elementary fibres together was removed by the

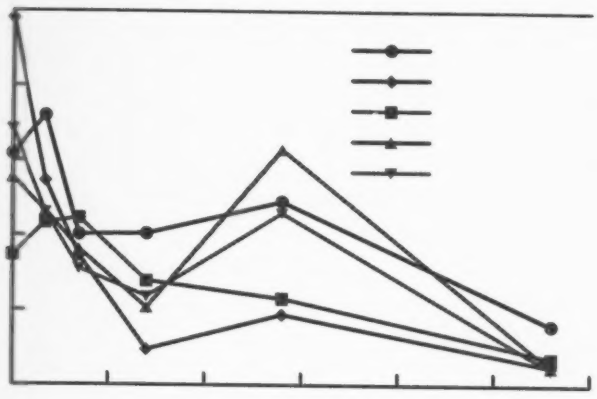


Figure 12. Change in elastic modulus with exposure time for all fibre treatments.

porewater, causing the technical fibre to break into its component elementary fibres. Initially, the technical fibre was able to act as a unit, and was therefore relatively strong and brittle. However, when the middle lamella was dissolved by the concrete porewater, the tensile strength decreased and the failure became less brittle as the elementary fibres were able to more easily slide relative to each other.

The measured values for the tensile properties of the untreated fibre prior to exposure to the porewater solution are significantly lower than those reported in the literature for flax fibre. For example, Bos et al. (2002) report tensile strengths of 1500 MPa for elementary fibres and at least 500 MPa for technical fibres. Published values for elastic modulus are in the range of 27 to 50 GPa (Bledzki and Gassan 1999, Bos and Donald 1999). Several factors may be responsible for this. First, it is important to recognize again that the fibres used for the current tension tests were technical fibres, consisting of bundles of elementary fibres (single plant cells) with a mean length of 30 μm (Bos et al. 2002) glued together by a middle lamella interface consisting of pectin, hemicellulose and lignin. Because the clamping length of 72 mm was longer than the length of individual elementary fibres, failure likely took place by shearing of the elementary fibres relative to each other through the middle lamella layer. Bos et al. (2002) and Bledzki and Gassan (1999) both report decreasing tensile strengths with increasing fibre lengths as a result of this effect; however, the strength is reported to level off near a value of 500 MPa for fibre lengths greater than approximately 25 mm. In this regard, it is instructive to note that the maximum tensile strengths for individual specimens measured as part of the current study were in the range of 450 to 600 MPa. In addition, published values for tensile properties are typically for textile fibre, rather than fibre obtained from oilseed flax plants, as was used for the current study. Finally, strength has also been found to be highly dependent on growing conditions and fibre processing technique (Jähn et al. 2002, Bos et al. 2002).

A significant variation in tensile strength was observed for fibre specimens with different diameters, with smaller diameter fibres exhibiting higher strengths. This can be explained by the well-known phenomenon of the increasing likelihood that critical defects will be present as the specimen size increases. In the context of the flax fibre, this

corresponds to an increasing probability of there being weaker areas in the bond between elementary fibres as the size of the technical fibre increased. This dependence is illustrated in Fig. 13, which plots the variation of mean tensile strength of the untreated flax fibre for fibres of all sizes and for fibres with diameters less than 150 μm. On average, the smaller fibres were 23% stronger, not including the fibres tested at 112 days, at which time the smaller diameter fibres were 176% stronger. A similar trend was observed for the elastic modulus, as well as for fibres with all different treatments. Data for all tests are tabulated in Appendix A.

The loss of strength and stiffness as a result of exposure to the porewater solution was significant for all fibres tested. However, this deterioration of properties may not necessarily be detrimental to the performance of concrete containing flax fibre. The primary reason for adding flax fibre to concrete is its ability to reduce plastic shrinkage cracking, which occurs within the first day after casting. For this application, the fibre has performed its intended function long before it is significantly affected by the concrete environment. In addition, if the reduction in tensile properties is the result of the technical fibres being separated into their component elementary fibres, as discussed above, the elementary fibres may remain intact and capable of functioning as a reinforcement.

3.1.4. Water Retention

Table 7 and Fig. 14 show the results of the water retention tests. The untreated fibre showed the least water retention among all fibre types, a result that was unexpected. All fibre treatments resulted in an immediate significant increase in water retention relative to the untreated fibre, with mercerization producing the greatest increase of 51%. Subsequent treatment with silane, acetylation, or acrylation reduced the water retention by 19 to 23% relative to the mercerized fibre, but the treated fibres' ability to retain water remained significantly greater than that of the untreated fibre. The advantage of the additional treatments, as compared with the mercerized fibre, was lost within the first two weeks of exposure, with all treated fibres exhibiting water retention values between 181 and 203% at

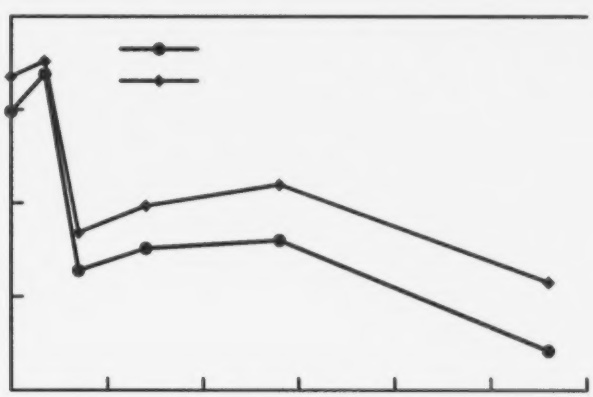


Figure 13. Influence of fibre diameter on measured tensile strength for untreated fibres.

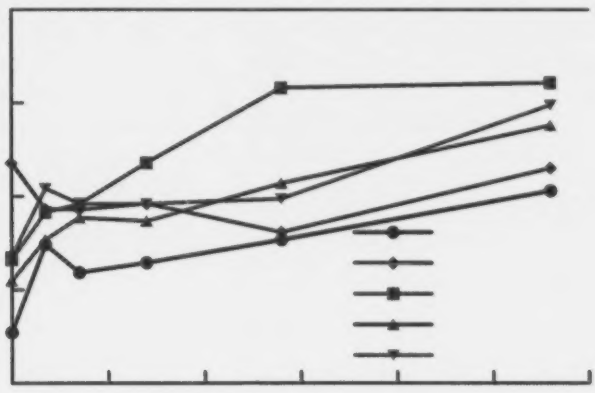


Figure 14. Change in water retention with exposure time for all fibre treatments.

Table 7. Water retention for all fibre treatments at all test intervals, as a percentage of dry mass.

Treatment	Exposure Time (days)					
	0	7	14	28	56	112
Untreated	142 (*6.6*)†‡	179 (*1.4*)	167 (*6.4*)	172 (*6.8*)	181 (*11*)	202 (*4.3*)
Mercerization	214 (*3.3*)	195 (*13*)	194 (*4.9*)	197 (*7.0*)	185 (*4.8*)	212 (*4.9*)
Silane	173 (*3.2*)	193 (*2.6*)	197 (*2.0*)	214 (*2.2*)	246 (*21*)	249 (*3.9*)
Acrylation	164 (*4.9*)‡	181 (*2.5*)	191 (*5.0*)	189 (*8.0*)	206 (*8.8*)	230 (*1.3*)‡
Acetylation	174 (*8.5*)	203 (*9.2*)	197 (*3.4*)	197 (*5.3*)	199 (*9.4*)	239 (*6.0*)

† The italicized values shown in parentheses are the coefficients of variation in percent.
‡ Indicates that an outlier was removed.

14 days. While the mercerized fibre maintained a more-or-less constant water retention for the duration of the tests, other fibre types experienced significant increases in water retention over 112 days of exposure. With the exception of the mercerized fibre, the three other treated fibre types maintained significantly higher water retention than the untreated fibre at 112 days.

When interpreting these results, it is important to distinguish water retention from moisture absorption. Moisture absorption refers to the accumulation of moisture from water vapour in the atmosphere, and reflects the accessibility of free hydroxyl groups for the formation of hydrogen bonds with water molecules (Pejic et al. 2008). On the other hand, water retention refers to ability to hold water in all water-retaining regions of the fibre after being soaked in liquid water. Because of these differences, water retention and moisture absorption can be affected differently by fibre treatments (Pejic et al. 2008). The durability of flax fibre in concrete is expected to be more influenced by its water retention properties, since the fibre will be in direct contact with porewater when embedded in concrete that is completely or partially saturated.

Pejic et al. (2008) reported that for hemp fibre, the selective removal of lignin, presumably from the middle lamella which binds individual elementary fibres together into a technical fibre bundle, resulted in a substantial increase in water retention. On the other

hand, the removal of hemicellulose, located primarily within the cell walls, resulted in a less pronounced decrease in water retention. In the current tests, the changes in the water retention properties caused by the chemical treatments appear to be dominated by the removal of lignin from the middle lamella, partially or completely separating elementary fibres and creating new spaces between the elementary fibres for the retention of water. The gradual unbonding the fibres over time by means of breaking down the middle lamella led to a gradual increase in water retention. None of the fibre treatments appears to have been effective in mitigating this degradation.

3.1.5. Scanning Electron Microscopy

Figure 15 shows a micrograph of an untreated individual elementary flax fibre prior to immersion in the porewater solution. Its diameter is just larger than 10 μm, and flakes of residual middle lamella material are visible on the fibre surface. In Fig. 16, an untreated technical flax fibre is shown after having been immersed in the porewater solution for 28 days. Here, individual elementary fibres are apparent, some still bonded together within the technical fibre bundle, and some separated. There is no obvious evidence of damage to the surfaces of the elementary fibres at this stage.

Several individual mercerized elementary fibres are seen in Fig. 17, after 7 days of immersion in porewater solution. These show evidence of separating along spiral lines at a small angle relative to the fibre axes, presumably following the path of cellulose meso-fibrils. The combination of mercerization and alkaline attack appears to have removed some of the hemicellulose matrix phase from the cell walls, causing the fibrils to separate. The appearance of the fibres did not differ from this at later stages. However, as seen in the micrographs of the following figures, mercerized fibres that were subsequently subjected to other treatments did not necessarily show evidence of similar damage to the elementary fibres. It is unlikely, therefore, that mercerization resulted in the damage to the elementary fibres seen in Fig. 17.

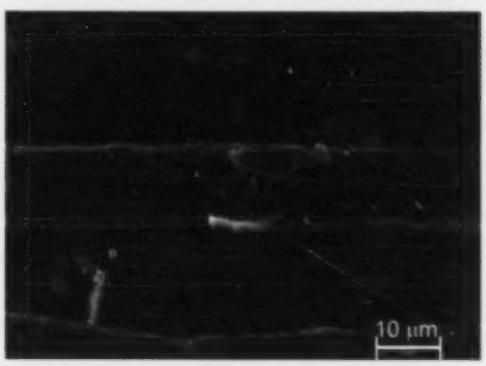


Figure 15. Scanning electron micrograph of an untreated elementary fibre prior to exposure to porewater solution.

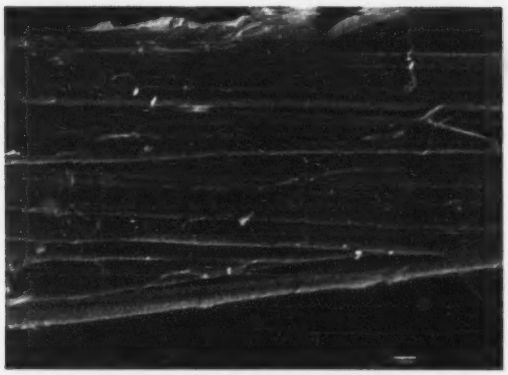


Figure 16. Scanning electron micrograph of an untreated technical fibre after 28 days of immersion in porewater solution.

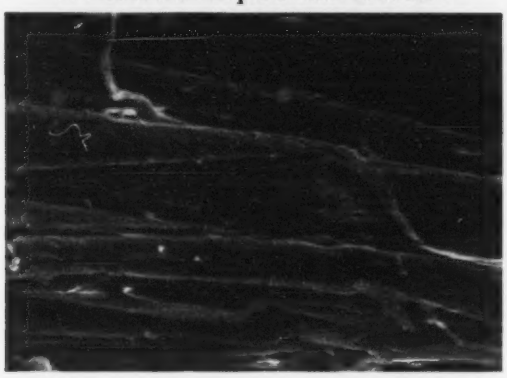


Figure 17. Scanning electron micrograph of mercerized elementary fibres after seven days of immersion in porewater solution.

Figure 18 shows a technical fibre bundle after silane treatment, but before being immersed in the porewater solution. Individual elementary fibres are visible within the bundle, with diameters ranging from 10 to 20 μm. The individual elementary fibre near the top of the frame is larger, with a diameter of approximately 35 μm, and appears to be in good condition. Another elementary fibre near the bottom of the frame, with a diameter of 20 μm, shows spirally oriented grooves on its surface, presumably following the path of underlying cellulose meso-fibrils.

Figure 19 shows acrylated fibre after 14 days of exposure. A number of elementary fibres, presumably once forming part of the same technical fibre bundle, are seen to be separated from each other at this location, and residuals of the middle lamella material are still apparent. However, there is no evidence of damage to the surfaces of the elementary fibres. After 56 days of exposure, the elementary fibres are still apparently in good condition (Fig. 20).

Figure 18. Scanning electron micrograph of a silane treated fibres prior to immersion in porewater solution.

Figure 19. Scanning electron micrograph of acrylated fibres after 14 days of immersion in porewater solution.

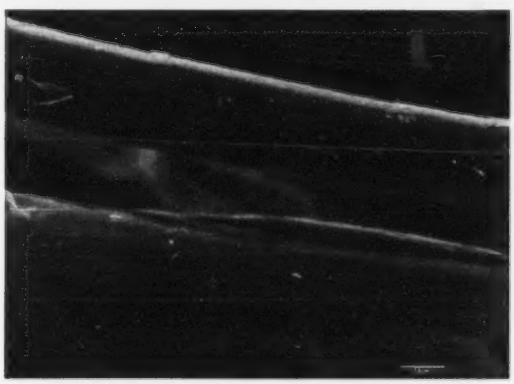


Figure 20. Scanning electron micrograph of acrylated elementary fibres after 56 days of immersion in porewater solution.

Figure 21 shows elementary acetylated fibres prior to immersion in porewater. Fibres appear to be splitting longitudinally. This damage may have been induced during processing, as micrographs taken at later stages of exposure do not show such obvious signs of damage. For example, Fig. 22 shows a relatively large elementary fibre after 28 days of exposure. It is mostly intact, although a small amount of splitting is apparent at the left of the frame. It is not clear whether this occurred due to processing or porewater exposure.

The SEM investigation shows that the technical fibres tended to separate into their constituent elementary fibres. This apparently occurred at several different stages, including the processing of the flax straw, the cleaning and removal of shives, the application of the chemical treatments, and the exposure to the highly alkaline porewater solution. None of the fibre treatments appears to prevent this separation. Although damage to the elementary fibres was apparent in some cases, its occurrence was not consistent and the cause can not be conclusively traced to any specific factor.

Figure 21. Scanning electron micrograph of acetylated fibres prior to immersion in porewater solution.

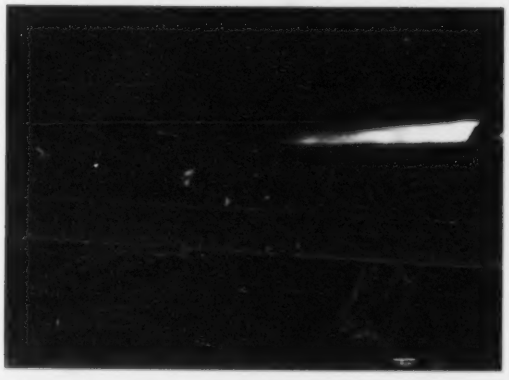


Figure 22. Scanning electron micrograph of acetylated fibres after 28 days of immersion in porewater solution.

3.1.6. Summary and Discussion of Phase 1 Results

To summarize, all fibre types lost mass as a result of their interaction with the synthetic porewater solution, with untreated fibre experiencing the greatest mass loss initially because the more soluble materials removed from treated fibres during mercerization were removed from the untreated fibre very early by alkaline attack of the porewater solution. All fibre types also experienced similar colour changes, as well as significant losses in their tensile properties (at least 80% in 112 days). The treated fibres were significantly weaker and less stiff than the untreated fibres after 112 days of exposure. Water retention increased for all fibre types during exposure, and the fibre treatments increased the water retention compared to the untreated fibres.

It is difficult to identify with certainty the mechanisms that would explain these observations. There are likely several interacting and competing factors at play. Based on the literature, two of these appear to dominate. The most important is the loss of bond that holds the elementary fibres together in technical fibre bundles, causing the elementary fibres to separate from each other. This occurs due to the dissolution of the pectin and lignin in the middle lamella, and is almost certainly responsible for the severe degradation of tensile properties with exposure time. This explanation is supported by the fact that it became increasingly difficult to obtain fibre samples of sufficient length and integrity at longer exposure times to perform tension tests. It is also consistent with the increase in water retention properties, as the removal of the middle lamella would create new spaces between the elementary fibres for the retention of water.

A second contributing factor could be the swelling and partial removal of hemicellulose from within the cell walls, making the matrix less dense, less rigid, and more uniform (Gassan and Bledzki 1999). This allows, firstly, a realignment of the cellulose meso-fibrils within the cell walls in the direction of applied tension, and, secondly, the formation of new hydrogen bonds between cellulose chains and increasing crystallization; both of these could lead to an increase in tensile properties. This could, for example, be responsible for the initial increase in the tensile properties with mercerization. The relatively long gauge length used for tension specimens meant that this effect could only be measured as long technical fibres remained intact, since the length of individual elementary fibres was significantly shorter than the gauge length.

Clearly, none of the chemical fibre treatments was effective at mitigating the degradation caused by exposure to the porewater solution. In fact, the untreated fibres were found to have superior tensile and water retention properties after 112 days of exposure. As a result, a new treatment was introduced for evaluation as part of Phase 2 of the project, as presented in the following section.

3.2. Phase 2 – Part 1 – Effect of Wet-Dry Cycling on Properties of FFRC

3.2.1. Compressive Strength

Compressive strength provides a measure of the general quality of a particular concrete, and allows a comparison of quality between different batches and different weathering scenarios. Measured compressive strengths for all specimen types and weathering scenarios for the wet-dry cycling tests are listed in Table 8. Here, it is noted that data labelled "Ambient 50" and "Ambient 25" correspond to the cylinders stored in ambient laboratory conditions and tested at the same time as those that underwent 50 wetting-drying cycles lasting four days each and 25 wetting-drying cycles lasting eight days each, respectively. The table shows that the 28 day compressive strengths for all specimen types were comparable, indicating an excellent level of consistency among the four different batches of concrete. The slightly higher strength of specimens with silane treated fibres reflects an expected level of variation among different concrete batches. It is generally accepted that the presence of fibres at such low dosages has an insignificant effect on the strength of concrete, either compressive or flexural (Folliard and Simpson 1998).

Table 8. Average compressive strengths (MPa) for cylinders tested as part of the wet-dry cycling study.

Fibre Treatment	Weathering scenario				
	28 day cure	50 cycles	25 cycles	Ambient 50*	Ambient 25*
Plain concrete	24.7 (*3.1*)†	24.8 (*1.9*)	24.0 (*1.3*)	31.1 (*3.7*)	33.0 (*3.7*)
Untreated fibre	25.2 (*5.9*)	25.1 (*7.4*)	25.6 (*4.4*)	33.7 (*5.9*)	32.0 (*3.9*)
Duralin	25.7 (*5.4*)	26.0 (*5.7*)	26.4 (*5.3*)	34.4 (*5.8*)	34.7 (*3.1*)
Silane	26.8 (*2.0*)	27.0 (*5.7*)	28.5 (*1.2*)	36.2 (*6.0*)	35.0 (*5.0*)

All values were determined from 4 replicate measurements.
† The italicized values shown in parentheses are the coefficients of variation in percent.
* Ambient 50 and Ambient 25 correspond to specimens stored in ambient laboratory conditions and tested at the same time as specimens subjected to 50 and 25 cycles, respectively.

In order to remove the influence of different 28-day strengths when studying the effect of the different weathering scenarios, mean compressive strengths for each specimen type were normalized by their respective 28-day values; the results are plotted in Fig. 23. Error bars correspond to standard deviations, which were also normalized by the respective 28-day strengths. The control behaviour, against which the performance of different specimen types may be compared, is shown by the plain concrete specimens. Neither of the two wet-dry cycling scenarios resulted in any significant changes to the compressive strength of the plain specimens. However, the compressive strengths of specimens cured under ambient laboratory conditions were 26 to 38% higher than their weathered counterparts. This is believed to be the result of the different moisture contents in these two groups of specimens; whereas specimens from all other weathering scenarios were tested in a saturated condition, those stored in ambient conditions were tested in an air-dry condition. It is known that a compression specimen tested in air dry conditions will be stronger than one tested in a saturated condition. While the increase in strength is generally believed to be in the order of 10 to 15% (Young et al. 1998, Mindess et al. 2003), increases as high as 40% have been reported (Troxell and Davis 1956, p. 191).

Figure 23 and Table 8 show that the behaviours of the other three specimen types, each containing flax fibre in different forms, were similar to that of the plain concrete specimens.

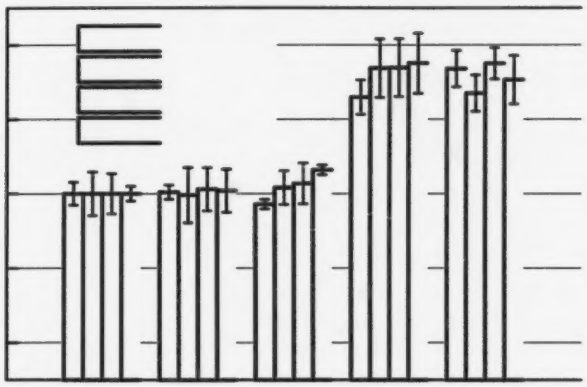


Figure 23. Average compressive strength of cylinders tested as part of the wet-dry cycling study.

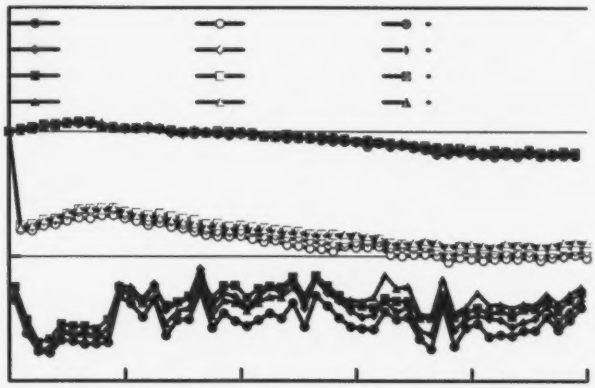
Although some of the differences in strength among different groups of specimens were statistically significant at the 90% confidence limit (e.g., specimens with silane treated fibres undergoing 25 eight-day cycles were slightly stronger than at 28 days), these differences tended to be relatively small. The laboratory cured specimens were between 23 and 34% stronger than their weathered counterparts. The similarities to the plain specimens indicate that the presence of fibres was not detrimental to the compressive strength of the concrete, regardless of the fibre treatment.

3.2.2. Dynamic Modulus of Elasticity

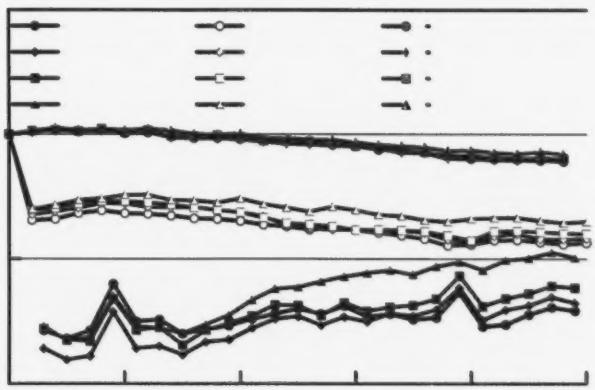
The dynamic modulus of elasticity is used to track changes to the mechanical properties of concrete over time using a non-destructive test. Figure 24 illustrates the change in elastic modulus with an increasing number of cycles for specimens subjected to 50 four-day wetting-drying cycles (Fig. 24a) and 25 eight-day wetting-drying cycles (Fig. 24b). Measurements were made at two different times during each wetting-drying cycle, once when specimens were in a wet condition, immediately after being removed from the water bath, and again in a dry condition, immediately after being removed from the drying oven. A significant difference between the two measurements is apparent, as is expected (Troxell and Davis 1956, p. 261).

The control specimens, left in ambient laboratory conditions, experienced a gradual reduction in elastic modulus over time, with a maximum reduction of approximately 4% over the 250 day weathering period. This is likely due to a gradual loss of moisture over time. No significant differences among specimen types can be observed.

For specimens subjected to wetting and drying, a significant loss of modulus was observed after the first cycle, on the order of 12 to 15% for specimens tested in a wet condition, and closer to 35% for specimens tested in a dry condition. This loss is likely a direct result of the first drying cycle, during which the microstructure of the cement paste would have been altered irreversibly as moisture was removed from micropores, allowing calcium-silicate-hydrate particles in the cement paste to shift closer together. Subsequently,



(a)



(b)

Figure 24. Change in dynamic elastic modulus with wet-dry cycles for (a) 50 four-day cycles, and (b) 25 eight-day cycles.

the wet specimens experienced a gradual loss in modulus, as each cycle would cause slight changes to the microstructure of the cement paste. The behaviour of specimens with fibres was similar to that of the plain specimens, suggesting that the changes experienced can not be attributed to the presence of fibres. The specimens tested in a dry condition exhibited greater variability. After the initial drop of approximately 35%, these specimens experienced a gradual recovery, the reason for which cannot be explained at present. These specimens showed greater separation between different fibre types. As the dynamic modulus was slightly higher for FFRC specimens relative to the plain concrete specimens, the presence of fibres do not appear to have been detrimental to the performance of the concrete. FFRC specimens with treated fibres, particularly the silane treatment, generally performed slightly better than other specimens.

3.2.3. Flexural Strength

Measured flexural strengths (also known as modulus of rupture) for all specimen types and weathering scenarios are listed in Table 9. Again, data labelled "Ambient 50" and "Ambient 25" correspond to results for the beams stored in ambient laboratory conditions and tested at the same time as those that underwent 50 four-day wet-dry cycles and 25 eight-day wet-dry cycles. The flexural strengths of all specimen types at 28 days are within the range of expected values generally observed for plain concrete with the compressive strengths listed in Table 8 (Kosmatka et al. 2002, p. 283). The 28-day values for all specimen types were relatively close; however, silane treated FFRC specimens were statistically stronger at the 90% level of significance. This is believed to be a reflection of the expected level of variation among different concrete batches.

Table 9. Modulus of rupture (MPa) for beams tested as part of the wet-dry cycling study.

Fibre Treatment	Weathering scenario				
	28 day cure	50 cycles	25 cycles	Ambient 50‡	Ambient 25‡
Plain concrete	3.87 (*3.7*)†	3.21* (*3.7*)	3.27 (*9.7*)	4.57 (*7.1*)	4.92 (*8.4*)
Untreated fibre	3.79 (*5.5*)	3.42 (*13*)	3.59 (*6.7*)	5.08 (*11*)	4.66 (*6.2*)
Duralin	3.62 (*9.2*)	3.20 (*22*)	3.52 (*18*)	4.53 (*9.7*)	4.51 (*6.3*)
Silane	4.20 (*4.1*)	3.24 (*16*)	3.71* (*12*)	4.98* (*4.6*)	4.35 (*6.7*)

Listed values are the average of 4 replicate measurements, except those marked by *, for which only 3 measurements were used because these beams failed outside of the middle third of their span.
† The italicized values shown in parentheses are the coefficients of variation in percent.
‡ Ambient 50 and Ambient 25 correspond to specimens stored in ambient laboratory conditions and tested at the same time as specimens subjected to 50 and 25 cycles, respectively.

Figure 25 plots the same data, with the flexural strengths for each specimen type normalized by their respective 28-day values in order to remove the influence of differences in 28-day strengths. This figure shows that the plain concrete specimens experienced a 15 to 17% drop in flexural strength as a result of wet-dry cycling, whereas the laboratory-cured specimens were 18 to 27% stronger. The specimens containing flax fibres generally followed similar trends. However, the losses after cycling were not statistically significant at the 90% level of significance for these specimens, except for those with silane treated fibres subjected to 50 four-day cycles. The increases experienced by the laboratory-cured FFRC specimens ranged between 18 and 34%, except, again, for one of the groups containing silane treated fibres. The inconsistent performance of the FFRC specimens with silane treated fibres, for which those subjected to 25 cycles and one of the laboratory-cured groups followed the trends of the other specimens while the other groups appeared to deviate from the trends, does not permit a conclusive evaluation of this treatment based on this set of tests. However, it is most likely that the silane treated specimens were similar to the others. The fact that FFRC specimens followed similar trends to plain specimens suggests that the presence of the fibres was not detrimental to the performance of the concrete.

3.2.4. Flexural Toughness

Figure 26 shows three typical load-deflection responses from the flexural tests performed on beams after the 28 day curing period. The areas representing the pre- and post-peak toughness values are also shown for the Duralin FFRC specimen to clarify how toughness was calculated. As represented by the response of the plain specimen shown, specimens without fibres failed immediately upon reaching the maximum load, at which

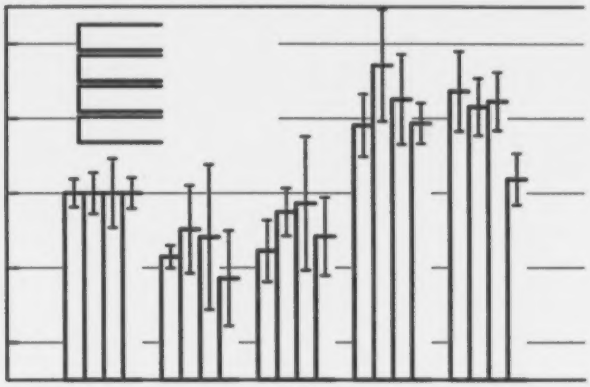


Figure 25. Average flexural strength of beams tested as part of the wet-dry cycling study.

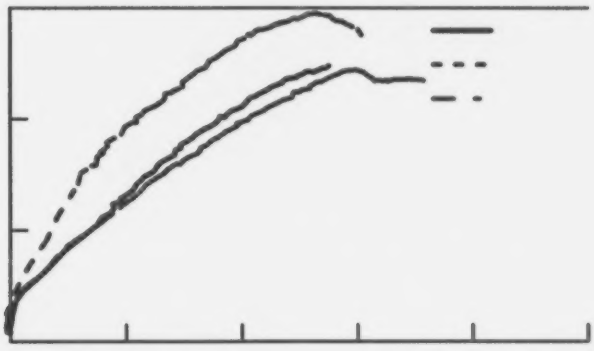


Figure 26. Typical load-deflection behaviours for beams tested at 28 days as part of the wet-dry cycling study.

point they cracked and broke into two pieces. In a fibre-reinforced specimen, fibres are expected to bridge this crack and provide some load-carrying capacity beyond the point of cracking. The responses of the silane and Duralin FFRC specimens show that the fibres did provide some capacity across the crack immediately after the peak load was reached; however, they were not able to sustain this capacity for long as the crack widened with increasing flexural deflections. This observation confirms the results of previous tests performed on specimens containing untreated flax fibre (Wang and Wegner 2001). As a result, post-peak toughness values for all FFRC specimens were relatively low.

The average toughness values calculated for all specimen types and weathering scenarios are listed in Table 10 and plotted in Fig. 27. In Fig. 27, the post-peak portion of the total toughness is shown stacked on top of the pre-peak portion as a visual representation of its contribution to total toughness. Although fibres did impart some post-peak toughness to

the concrete, the relatively low post-peak toughness values indicate that the contribution of any of the fibre types was small. Post-peak toughness typically accounted for between 10 and 30% of the total toughness of FFRC specimens. Neither the fibre treatment nor wet-dry cycling effectively changed the performance of the fibres in this regard. This finding indicates that flax fibre should not be considered as a method to improve the flexural toughness of concrete, at least not until a fibre treatment effective at reducing the bond strength is found. However, the presence of the flax fibre did not negatively influence the properties of the concrete subjected to wet-dry cycling.

Table 10. Flexural toughness (Joules) for beams tested as part of the wet-dry cycling study.

Fibre Treatment	Weathering scenario				
	28 day cure	50 cycles	25 cycles	Ambient 50‡	Ambient 25‡
Plain concrete					
Pre-peak	0.16	0.09	0.10	0.16	0.14
Post-peak	0.00	0.00	0.00	0.00	0.00
Total	0.16 (*29*)†	0.09* (*21*)	0.10 (*59*)	0.16 (*26*)	0.14 (*7.1*)
Untreated fibre					
Pre-peak	0.17	0.13	0.16	0.26	0.16
Post-peak	0.04	0.03	0.05	0.03	0.02
Total	0.21 (*32*)	0.17 (*36*)	0.20 (*29*)	0.28 (*17*)	0.18 (*31*)
Duralin					
Pre-peak	0.18	0.13	0.13	0.23	0.16
Post-peak	0.03	0.02	0.05	0.02	0.05
Total	0.21 (*45*)	0.15 (*51*)	0.18 (*69*)	0.26 (*32*)	0.21 (*45*)
Silane					
Pre-peak	0.24	0.12	0.17	0.23	0.17
Post-peak	0.03	0.02	0.05	0.04	0.07
Total	0.27 (*15*)	0.14 (*33*)	0.21* (*5.5*)	0.27* (*26*)	0.24 (*42*)

Listed values are the average of 4 replicate measurements, except those marked by *, for which only 3 measurements were used because these beams failed outside of the middle third of their span.

† The italicized values shown in parentheses are the coefficients of variation in percent.

‡ Ambient 50 and Ambient 25 correspond to specimens stored in ambient laboratory conditions and tested at the same time as specimens subjected to 50 and 25 cycles, respectively.

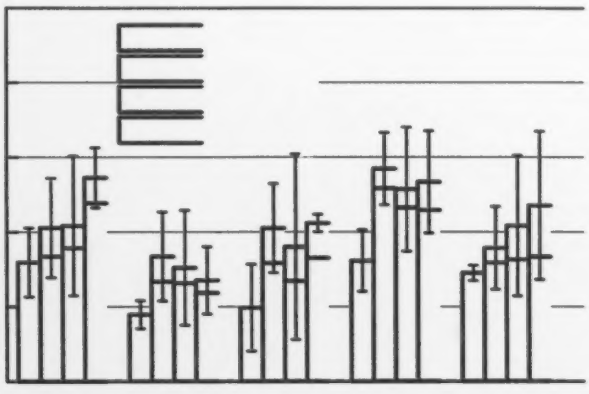


Figure 27. Average flexural toughness of beams tested as part of the wet-dry cycling study. The post-peak portion of the total toughness is shown stacked on the pre-peak portion for each group.

3.2.5. SEM Observation of Fibres on Failure Surfaces

SEM observations of the failure surfaces of beam specimens revealed only elementary fibres. It appears that any technical fibres that may have been incorporated into the fresh concrete mix had separated into their component elementary fibres during mixing.

Figure 28 shows SEM micrographs of untreated flax fibres observed on the failure surfaces of beam specimens that had been subjected to different weathering scenarios. Figure 28a, taken after 28 days of moist curing, shows a fibre coated with cement paste, while the other two micrographs are less coated. This does not suggest, however, that the wetting-drying cycles influenced the bond of cement paste to fibre, as there was no generally observed correlation between the two. In fact, the micrographs shown in this and subsequent figures represent several different conditions encountered (e.g., some coated with cement paste, some with fibrils apparently separating from the main body of a fibre, some longer or shorter, etc.), but no correlation between any of these features and a particular fibre treatment or weathering scenario was observed.

Figures 29 and 30 show SEM micrographs of Duralin and silane treated fibres, respectively, again for different weathering scenarios. Again, no obvious differences were observed among different fibre types or weathering scenarios.

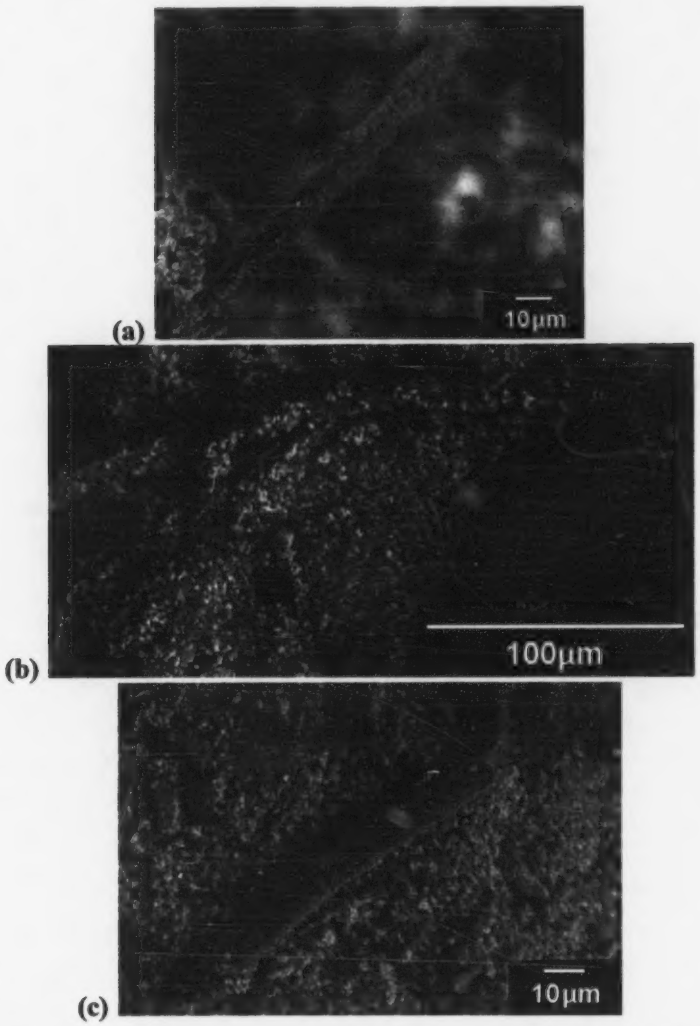


Figure 28. SEM micrographs of untreated flax fibres taken on beam failure surfaces (a) after 28 days of moist curing, (b) after 50 four-day wetting-drying cycles, and (c) after 25 eight-day wetting-drying cycles.

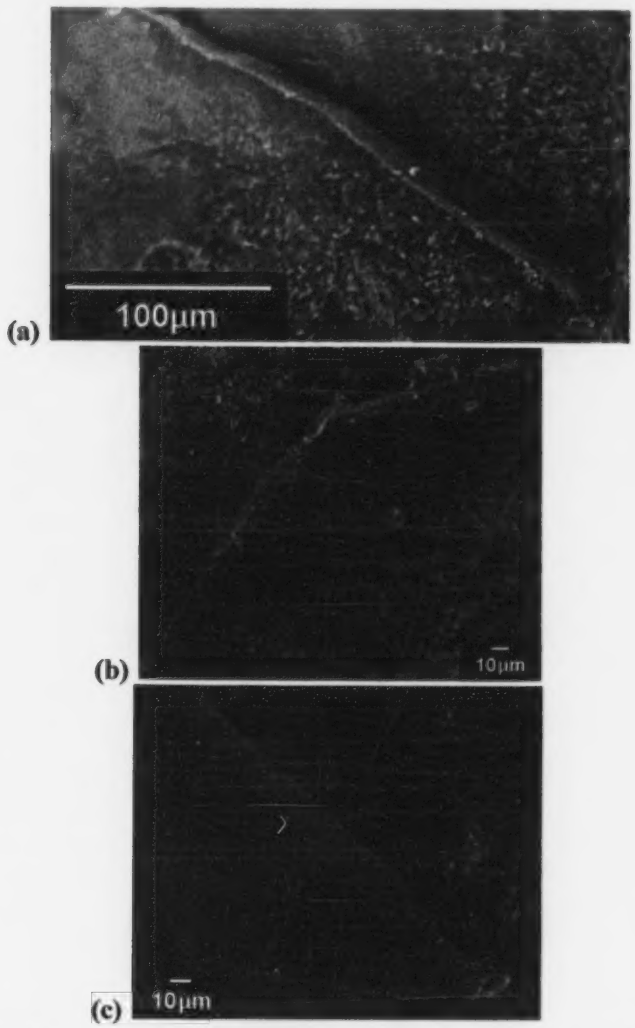


Figure 29. SEM micrographs of Duralin treated flax fibres taken on beam failure surfaces (a) after 28 days of moist curing, (b) after 50 four-day wetting-drying cycles, and (c) after 25 eight-day wetting-drying cycles.

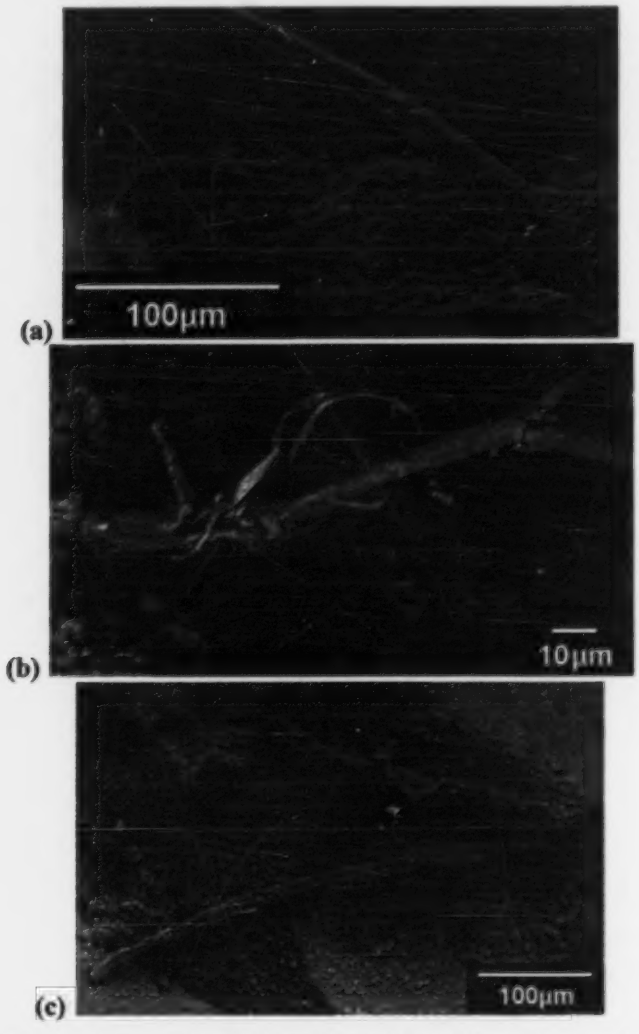


Figure 30. SEM micrographs of silane treated flax fibres taken on beam failure surfaces (a) after 28 days of moist curing, (b) after 50 four-day wetting-drying cycles, and (c) after 25 eight-day wetting-drying cycles.

3.2.6. Chemical Composition of Fibres

Figure 31 shows the elemental analysis of the cement paste. It consists of the expected elements, dominated by calcium and silicon. In Fig. 32, the elemental analyses of the three types of fibre, prior to exposure to concrete, are shown. Some differences in chemical composition are seen for the fibres chemically treated with silane. However, the elemental analyses of the fibre surfaces after being embedded in concrete and subjected to 50 wetting-drying cycles over 250 days, as seen in Fig. 33, show that calcium became much more prominent, and that the surfaces of all three fibre types featured very similar chemical compositions. This essentially shows that the surface of the fibres took on the chemical composition of the cement paste after having been embedded in it for a lengthy time period. It should be noted that this analysis was limited to the surface of the fibres, so it is not possible to draw definite conclusions about the mineralization of the interior of the fibres.

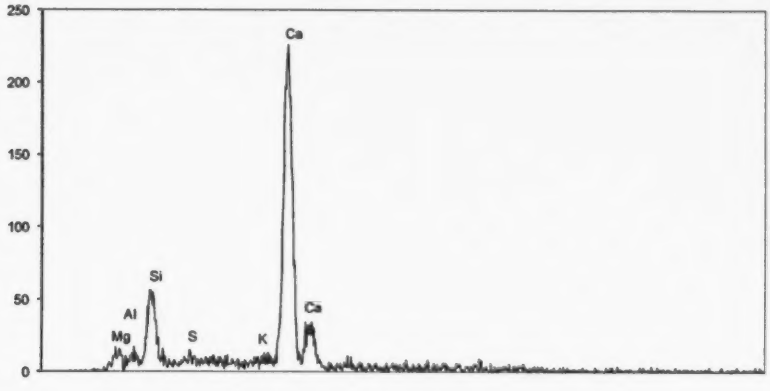


Figure 31. Elemental analysis of cement paste.

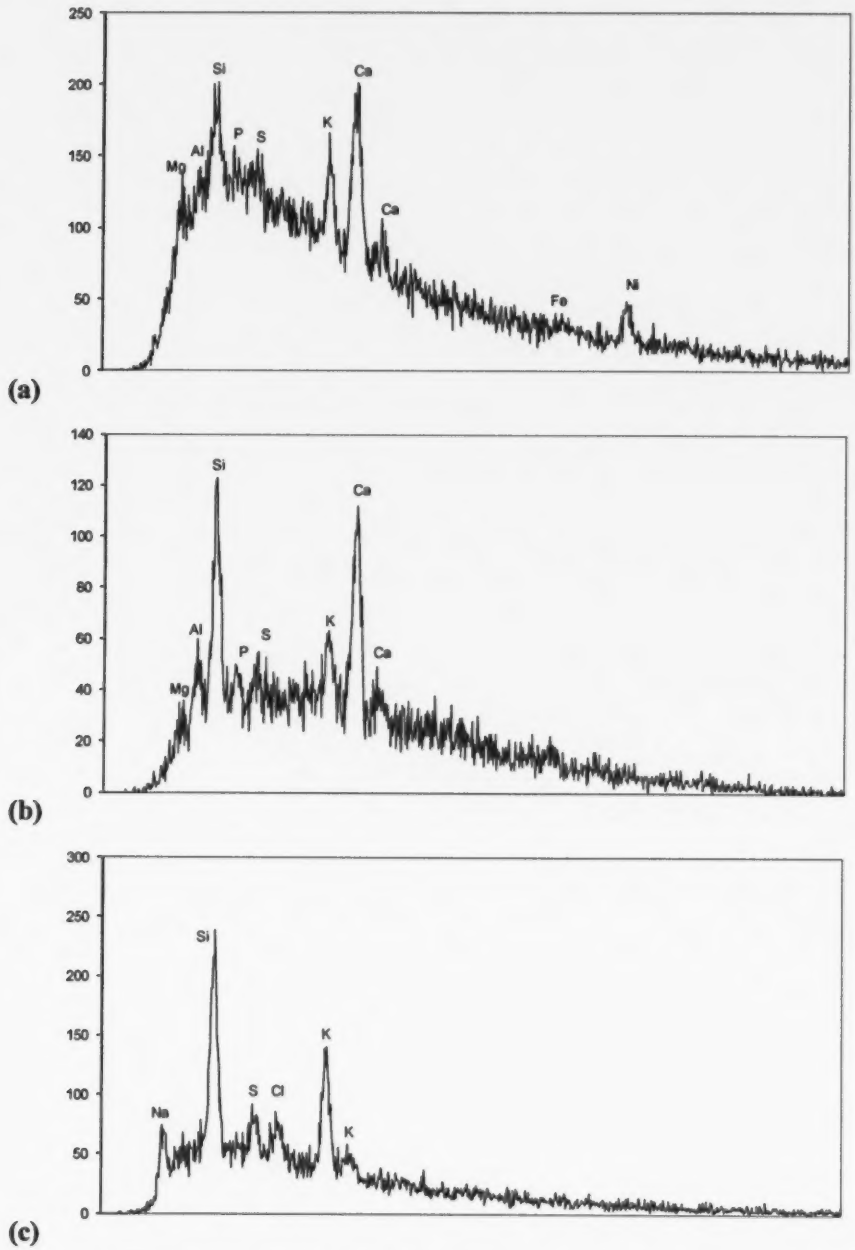


Figure 32. Elemental analysis of flax fibre that was not mixed into the concrete, in (a) untreated, (b) Duralin treated, and (c) silane treated forms.

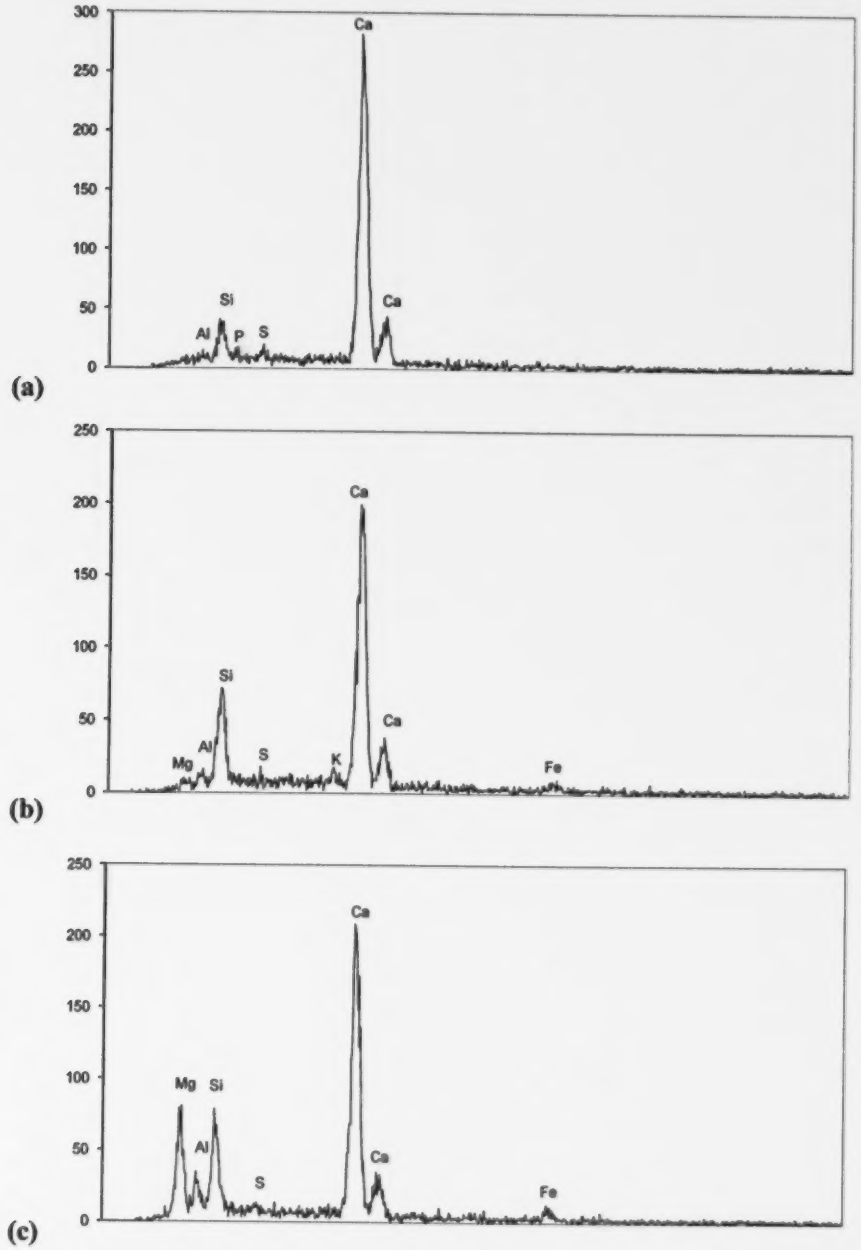


Figure 33. Elemental analysis of exposed flax fibres on the failure surfaces of beams subjected to 50 four-day cycles of wetting and drying, in (a) untreated, (b) Duralin treated, and (c) silane treated forms.

3.2.7. Summary and Discussion of Wet-Dry Cycling Study

To summarize, the presence of flax fibres did not have a significant influence on the compressive properties of concrete, regardless of fibre treatment or wetting-drying scenario. As compressive strength is routinely used as an indicator of the overall quality of concrete, this suggests that the mechanical properties of FFRC subjected to cyclic wetting and drying will not be affected any differently than concrete without fibres. These results were confirmed by measurements of the dynamic elastic modulus and flexural strength, for which FFRC specimens followed trends that were similar to those of plain concrete.

Flexural toughness tests confirmed the results of earlier work, which found that flax fibre was not effective at imparting a significant level of flexural toughness to concrete. The contribution of fibres is therefore not sufficient for use in applications that require an increase in flexural toughness. Neither of the two fibre treatments studied, nor exposure to repeated wetting and drying cycles, influenced this result significantly. However, the presence of the fibres did not negatively influence the toughness properties of the concrete, even after 50 four-day wetting-drying cycles.

Microscopic examination using a scanning electron microscope could find no obvious visible changes to the fibres as a result of treatment or cyclic wetting and drying. However, the surface of the fibres was found to gradually take on the chemical composition of the cement paste.

3.3. Phase 2 – Part 2 – Effect of Freeze-Thaw Cycling on Properties of FFRC

3.3.1. Compressive Strength

The mean compressive strengths of samples tested as part of the freeze-thaw study are listed in Table 11. It should be recalled that both cylinder and block specimens were tested at 28 days, but that only block specimens were tested for the other batches due to the inability of the freeze-thaw chamber to accommodate cylinders. The results of the block tests were therefore used to ascertain the influence of freeze-thaw cycling on the compressive strength. It was observed, though, that the standard cylinder specimens tested at 28 days were slightly weaker than the companion non-standard blocks; this difference was only statistically significant for the silane treated FFRC. Although not shown here, in 80% of the cases tested for the wet-dry cycling study, there was no significant difference between the compressive strengths of cylinders and blocks.

Table 11 shows that there was a large variation in strength among the four different materials tested. In fact, the untreated FFRC specimens were roughly twice as strong as those containing the silane treated fibre, and exceeded the capacity of the test machine (a fact that was responsible for the low coefficients of variability for the untreated FFRC specimens). The differences in strength may be attributed to the differences in air content among the four batches. A comparison with Table 2 shows that the strength values correlate strongly with air content, with those containing the least air (untreated) exhibiting the highest strength, and those with the most air (silane treated) exhibiting the lowest strength. This observation is also consistent with previous findings (Kosmatka et al. 2002, p. 137).

Table 11. Average compressive strengths (MPa) for cylinders and block specimens tested as part of the freeze-thaw cycling study.

Fibre Treatment	Weathering scenario			
	28 day (cylinders)	28 day (blocks)	300 cycles	Ambient‡
Plain concrete	39.5 (*1.8*)†	41.5 (*9.6*)	36.9 (*8.2*)	47.3 (*9.5*)
Untreated fibre	59.8 (*2.0*)	>63.2£ (*1.9*)	>62.8£ (*0.4*)	>64.0£ (*0.4*)
Duralin	43.4 (*12*)	45.2 (*12*)	45.0 (*6.3*)	50.9* (*10*)
Silane	28.5 (*8.0*)	32.6* (*8.1*)	28.5 (*8.1*)	32.7 (*8.0*)

Tabulated results represent the mean values of 4 replicate measurements for cylinder tests, or 8 replicate measurements for block tests, except for values indicated by *, for which 6 or 7 replicate measurements were used.

† The italicized values shown in parentheses are the coefficients of variation in percent.

‡ Ambient specimens were stored in ambient laboratory conditions and tested at the same time as specimens subjected to 300 freeze-thaw cycles.

£ Maximum load of test machine was reached. Specimens did not fail.

In order to remove the influence of strength differences among the different materials to facilitate comparisons, strengths were normalized by the corresponding 28 day strengths of block specimens. Results are plotted in Fig. 34. Again, the plain specimens may be used as a benchmark against which to compare the performance of the three materials containing flax fibre. Plain specimens experienced an 11% loss of strength after being subjected to 300 freeze-thaw cycles. The companion specimens cured under ambient laboratory conditions, on the other hand, were 14% stronger, although this increase was not statistically significant at the 90% level of confidence. Although the graph appears to show that specimens with untreated fibres were unaffected by the weathering scenario, this simply reflects that fact that these specimens reached the maximum load of the test machine and did not fail; no conclusion regarding the performance of this material can therefore be drawn. Specimens with Duralin treated fibres experienced no reduction in strength after freeze-thaw cycling, and an increase in strength similar to that of the plain specimens when cured under ambient conditions. Finally, freeze-thaw cycling led to a strength loss of 12% for the silane treated FFRC specimens, a level similar to the plain specimens. This is likely reflective of the

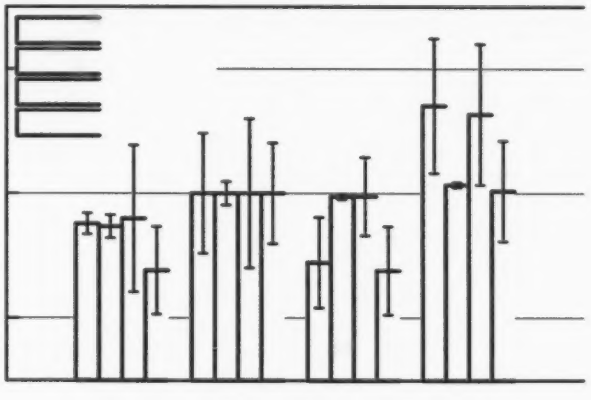


Figure 34. Average compressive strength of cylinders tested as part of the freeze-thaw cycling study, normalized by 28 day strengths of blocks.

relatively low entrained air content for these specimens. None of the FFRC specimens performed worse than the plain specimens after freeze-thaw cycling.

3.3.2. Dynamic Modulus of Elasticity

Due to irregularities in the test procedure followed by staff at the Lafarge laboratory, the dynamic modulus of elasticity (DME) was not measured at regular intervals during the freeze-thaw tests, as would normally be done, and values were therefore only available at the end of the three weathering scenarios. Results are tabulated in Table 12, and values normalized by their respective 28 day moduli are plotted in Fig. 35.

Table 12. Average dynamic modulus of elasticity (GPa) for specimens tested as part of the freeze-thaw cycling study.

Fibre Treatment	Weathering scenario		
	28 days	300 cycles	Ambient‡
Plain concrete	36.6 (*2.2*)	33.9 (*1.0*)	34.9 (*1.3*)
Untreated fibre	45.0 (*1.5*)	44.7 (*2.1*)	44.0 (*1.7*)
Duralin	37.4 (*1.3*)	36.0 (*0.8*)	37.5 (*1.1*)
Silane	30.8 (*1.3*)	27.3 (*2.2*)	29.6 (*1.6*)

Tabulated results represent the mean values of 4 replicate measurements.
† The italicized values shown in parentheses are the coefficients of variation in percent.
‡ Ambient specimens were stored in ambient laboratory conditions and tested at the same time as specimens subjected to 300 freeze-thaw cycles.

Similar to the compressive strength results, untreated FFRC specimens had significantly higher DME values both at 28 days and after weathering, while silane FFRC specimens had significantly lower values. Again, these variations are attributable to differences in air content.

Figure 35 shows that the control plain concrete specimens lost 5% of their original stiffness after experiencing 300 freeze-thaw cycles, reflecting a deterioration of the mechanical properties. Specimens cured in ambient laboratory conditions also lost approximately 7% of their original stiffness, but this likely reflects the lower moisture content of the ambient specimens at the time of testing. In contrast, the untreated and Duralin FFRC specimens experienced insignificant losses in properties after 300 freeze-thaw cycles, while the silane treated FFRC specimens lost 4% of their initial stiffness after freeze-thaw cycling, a loss similar to that of the plain control specimens. Losses in DME values after 90 days of curing in ambient laboratory conditions are thought to reflect the dryer state of these specimens.

FFRC specimens performed at least as well as the control plain specimens. Thus, the presence of fibre was not detrimental to the long-term durability of the concrete when subjected to freezing and thawing. In addition, the fibre treatments did not improve the performance relative to that of the specimens containing untreated flax fibre.

3.3.3. Flexural Strength

Measured flexural strengths (modulus of rupture) for specimens tested as part of the freeze-thaw cycling study are listed in Table 13. Again, relatively large differences between the various materials are apparent at 28 days, with untreated FFRC specimens stronger than the others and silane treated FFRC specimens weaker. On average, the flexural strengths

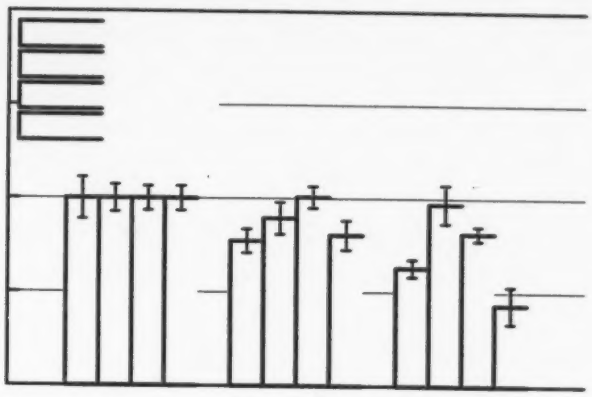


Figure 35. Average dynamic modulus of elasticity of specimens tested as part of the freeze-thaw cycling study, normalized by 28 day moduli.

Table 13. Modulus of rupture (MPa) for beams tested as part of the freeze-thaw cycling study.

Fibre Treatment	Weathering scenario		
	28 days	300 cycles	Ambient‡
Plain concrete	4.50 (*13*)†	5.90 (*5.0*)	6.74 (*5.3*)
Untreated fibre	6.43 (*3.3*)	7.27 (*2.1*)	8.47 (*5.8*)
Duralin	5.50 (*6.5*)	6.12* (*2.0*)	7.55 (*3.2*)
Silane	4.48 (*3.3*)	4.61 (*4.2*)	5.52 (*4.9*)

Tabulated results represent the mean values of 4 replicate measurements, except those marked by *, for which only 3 measurements were used.

† The italicized values shown in parentheses are the coefficients of variation in percent.

‡ Ambient specimens were stored in ambient laboratory conditions and tested at the same time as specimens subjected to 300 freeze-thaw cycles.

were approximately 30% higher than would normally be expected for concrete with the compressive strengths reported in Section 3.3.1. This finding cannot be explained at present.

Flexural strengths, normalized by their respective 28 day strengths to remove the influence of initial strength, are plotted in Fig. 36. As shown, the flexural strength of the plain control specimens increased significantly after freeze-thaw cycling and ambient curing, by 31% and 50%, respectively. This result was not expected. All specimens containing flax fibre in their various forms experienced more modest increases in flexural strength after freeze-thaw cycling and ambient curing, suggesting that the presence of the fibre is not detrimental to the performance of the concrete. The higher strengths of the ambient cured specimens likely reflect the air-dry condition of these specimens.

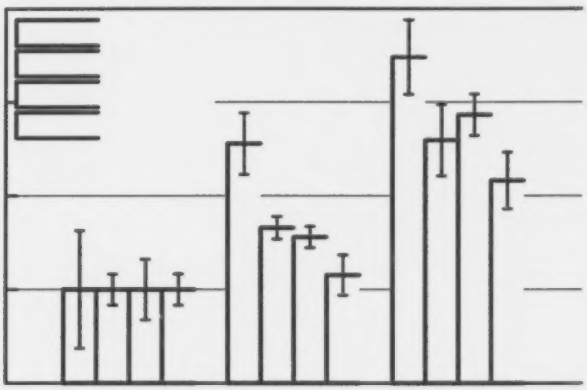


Figure 36. Average flexural strength of specimens tested as part of the freeze-thaw cycling study.

3.3.4. Flexural Toughness

Beams tested for flexural toughness as part of the freeze-thaw cycling study followed similar behaviours to those described in Section 3.2.4 and illustrated in Fig. 26; plain specimens failed immediately after cracking, whereas the fibres provided a very small amount of load-carrying capacity after initial cracking in fibre-reinforced specimens, resulting in very low post-peak toughness values. Results are listed in Table 14 and plotted in Fig. 37. Post-peak toughness accounted for between one and 24% of total toughness, but typically less than 10%. Although the presence of untreated and Duralin treated fibres improved the flexural toughness of the concrete at 28 days, further ambient curing and freeze-thaw cycling eliminated this improvement. Freeze-thaw cycling did not improve the flexural toughness of FFRC beams, as was hypothesized could occur as a result of a weakening of the bond between the fibre and cement paste. However, the presence of fibre was not detrimental to the performance of the concrete. Moreover, neither fibre treatment significantly improved the performance relative to specimens with untreated fibre.

3.3.5. SEM Observation of Fibres on Failure Surfaces

Figures 38 and 39 show representative SEM micrographs of the three types of fibres on the failure surfaces of beams tested in flexure at 28 days and after 300 freeze-thaw cycles, respectively. No differences among fibre types, or before and after weathering, are apparent. Examination of the failure surfaces revealed no evidence of fibre pullout from the cement paste, and the short protruding lengths of fibres from the failure surfaces suggested that fibre rupture was the dominant mode of failure. This is consistent with the low values for flexural toughness presented in the previous section and with previous findings for untreated flax fibre reinforced concrete specimens (Wang and Wegner 2001). The bond between fibres and cement paste matrix therefore remained intact, and fibres ruptured before cracks experienced substantial widening. Neither of the two fibre treatments nor freeze-thaw cycling appears to have affected the fibre-paste bond strength significantly.

Table 14. Flexural toughness (Joules) for beams tested as part of the freeze-thaw cycling study.

Fibre Treatment	Weathering scenario		
	28 day cure	300 cycles	Ambient‡
Plain concrete			
Pre-peak	0.21	0.32	0.47
Post-peak	0.00	0.00	0.00
Total	0.21 (*24*)†	0.32 (*21*)	0.47 (*14*)
Untreated fibre			
Pre-peak	0.37	0.29	0.35
Post-peak	0.12	0.01	0.03
Total	0.49 (*23*)	0.30 (*17*)	0.37 (*55*)
Duralin			
Pre-peak	0.34	0.34	0.40
Post-peak	0.02	0.01	0.00
Total	0.35 (*22*)	0.35* (*12*)	0.40 (*36*)
Silane			
Pre-peak	0.23	0.22	0.30
Post-peak	0.03	0.01	0.00
Total	0.26 (*13*)	0.23 (*36*)	0.30 (*28*)

Listed values are the average of 4 replicate measurements, except those marked by *, for which only 3 measurements were used because these beams failed outside of the middle third of their span.
† The italicized values shown in parentheses are the coefficients of variation in percent.
‡ Ambient specimens were stored in ambient laboratory conditions and tested at the same time as specimens subjected to 300 freeze-thaw cycles.

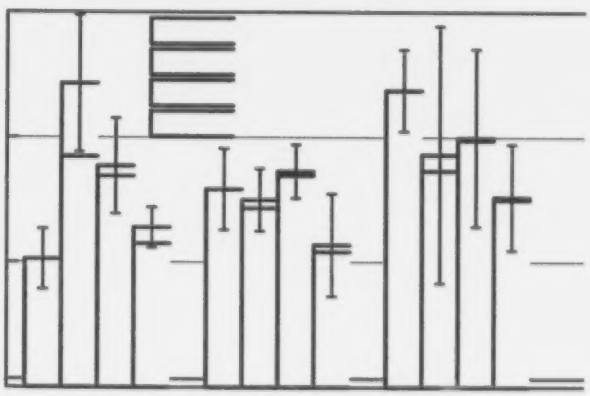
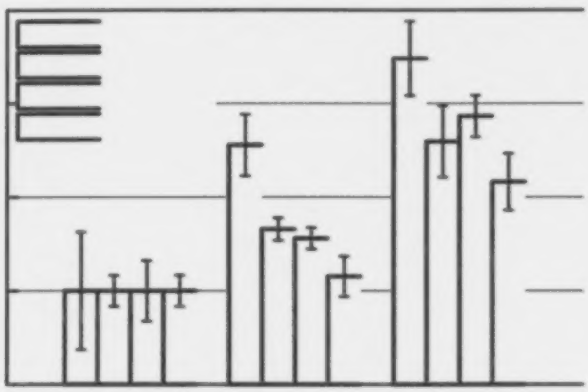


Figure 37. Average flexural toughness of beams tested as part of the freeze-thaw cycling study. The post-peak portion of the total toughness is shown stacked on the pre-peak portion for each group.



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Figures 38 and 39 show representative SEM micrographs of the three types of fibres on the failure surfaces of beams tested in flexure at 28 days and after 300 freeze-thaw cycles, respectively. No differences among fibre types, or before and after weathering, are apparent. Examination of the failure surfaces revealed no evidence of fibre pullout from the cement paste, and the short protruding lengths of fibres from the failure surfaces suggested that fibre rupture was the dominant mode of failure. This is consistent with the low values for flexural toughness presented in the previous section and with previous findings for untreated flax fibre reinforced concrete specimens (Wang and Wegner 2001). The bond between fibres and cement paste matrix therefore remained intact, and fibres ruptured before cracks experienced substantial widening. Neither of the two fibre treatments nor freeze-thaw cycling appears to have affected the fibre-paste bond strength significantly.

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Pre-peak	0.21	0.32	0.47
Post-peak	0.00	0.00	0.00
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Untreated fibre			
Pre-peak	0.37	0.29	0.35
Post-peak	0.12	0.01	0.03
Total	0.49 (*23*)	0.30 (*17*)	0.37 (*55*)
Duralin			
Pre-peak	0.34	0.34	0.40
Post-peak	0.02	0.01	0.00
Total	0.35 (*22*)	0.35* (*12*)	0.40 (*36*)
Silane			
Pre-peak	0.23	0.22	0.30
Post-peak	0.03	0.01	0.00
Total	0.26 (*13*)	0.23 (*36*)	0.30 (*28*)

Listed values are the average of 4 replicate measurements, except those marked by *, for which only 3 measurements were used because these beams failed outside of the middle third of their span.

† The italicized values shown in parentheses are the coefficients of variation in percent.

‡ Ambient specimens were stored in ambient laboratory conditions and tested at the same time as specimens subjected to 300 freeze-thaw cycles.

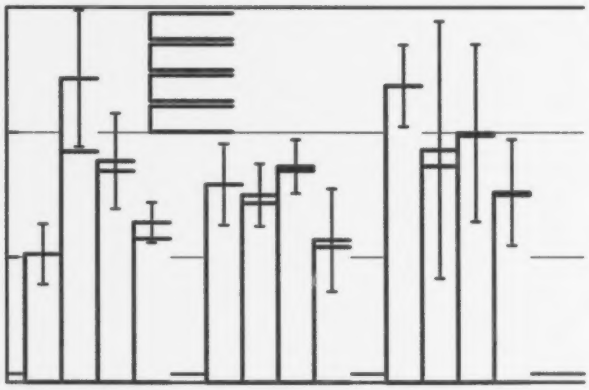

Figure 37. Average flexural toughness of beams tested as part of the freeze-thaw cycling study. The post-peak portion of the total toughness is shown stacked on the pre-peak portion for each group.

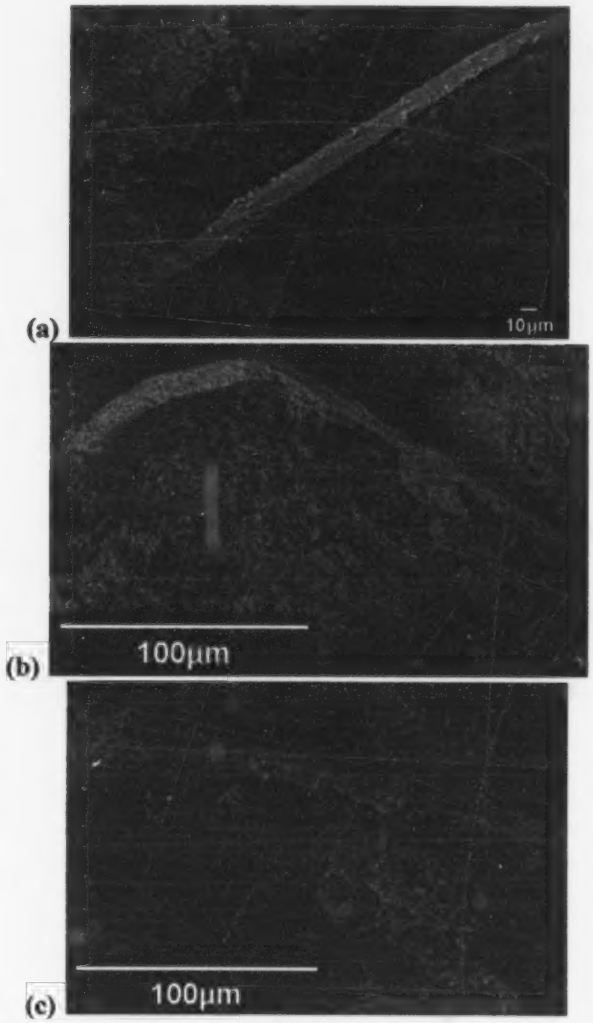

Figure 38. SEM micrographs of flax fibres taken on beam failure surfaces after 28 days of moist curing: (a) untreated, (b) Duralin treated, and (c) silane treated fibres.

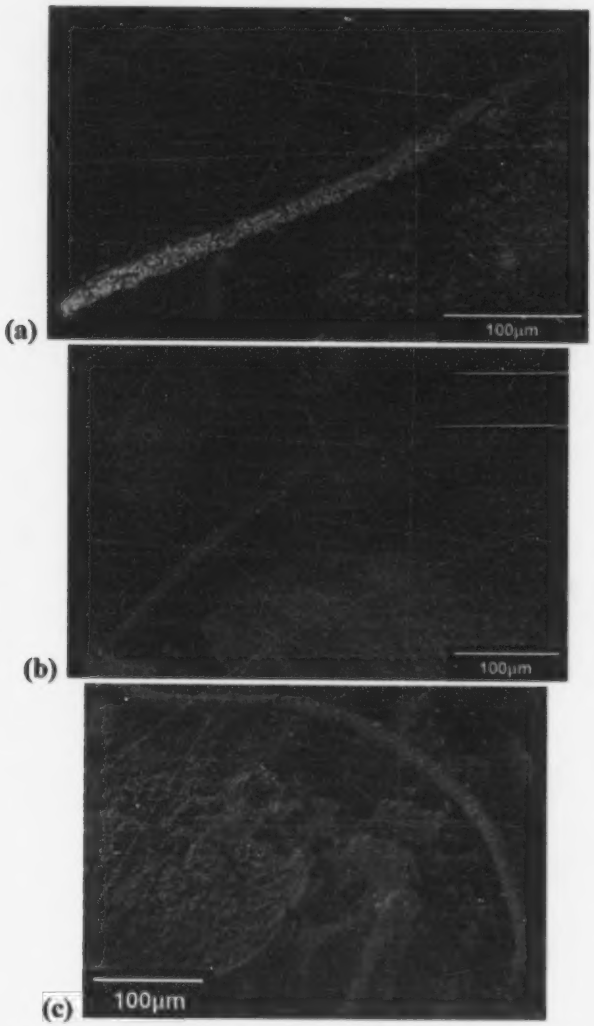

Figure 39. SEM micrographs of flax fibres taken on beam failure surfaces after 300 freeze-thaw cycles: (a) untreated, (b) Duralin treated, and (c) silane treated fibres.

3.3.6. Summary and Discussion of Freeze-Thaw Cycling Study

In summary, the presence of flax fibres did not negatively influence the mechanical properties of concrete relative to plain concrete after exposure to 300 rapid freeze-thaw cycles. This was demonstrated by measurements of compressive strength, dynamic modulus of elasticity, and flexural strength. Exposure to freeze-thaw cycles had no significant effect on the compressive strength or modulus of elasticity of concrete containing untreated and Duralin treated flax fibre. Silane treated FFRC specimens did not perform as well as the other two types of FFRC, but this is likely due to the inadvertent high entrained air content (12%) of these specimens. In all cases, all three FFRC materials performed at least as well as plain concrete after exposure to freeze-thaw cycling. Moreover, neither of the fibre treatments resulted in a significant improvement in the performance of the FFRC relative to specimens with untreated flax fibre.

Flexural toughness tests confirmed that the flax fibre was not capable of adding a significant level of toughness to the concrete due to the fact that fibres rupture at very small crack widths such that they do not provide continued load-carrying capacity as the crack widens. The high strength of the fibre-cement paste bond does not permit the fibres to pull out and dissipate energy. Neither of the two fibre treatments appears to have influenced the bond strength, and therefore the flexural toughness performance was not improved by fibre treatment. Furthermore, exposure to 300 freeze-thaw cycles also did not have a significant influence on the bond strength.

4. Conclusions and Recommendations

Phase 1 of this study showed that immersion of flax fibre in a highly alkaline synthetic cement porewater solution resulted in severe degradation of the fibre, apparently caused by the dissolution of the middle lamella that holds elementary fibres together in bundles that form the larger technical fibres. As a result, the technical fibres separated into their component elementary fibres, leading to an 86% loss of tensile strength after 112 days of immersion, since the grip length used for tension tests was significantly larger than the length of individual elementary fibres. Fibre separation also led to a 23% mass loss and 42% increase in the ability of the fibre to hold water.

None of the four fibre treatments evaluated in Phase 1 (including mercerization, silane, acrylation, and acetylation) improved the resistance of the flax fibre to chemical attack by the highly alkaline solution. Treated technical fibres lost 82 to 96% of their tensile strengths after 112 days of exposure to the porewater solution, and retained even more water than their untreated counterparts after 112 days of immersion. Thus, none of the chemical treatments was effective at protecting the fibre from degradation due to alkali attack.

Although the fibres themselves deteriorated and broke down into elementary fibres when exposed to synthetic porewater solution, Phase 2 of this study showed that incorporating flax fibre into concrete did not negatively affect the properties of the concrete after exposure to repeated wet-dry cycles over 250 days, or freeze-thaw cycles over 90 days. Changes to the compressive strength, dynamic modulus of elasticity, and flexural strength of concrete specimens containing flax fibre did not differ significantly from those of plain concrete specimens, after exposure to 50 wet-dry cycles and 300 rapid freeze-thaw cycles. Flax fibre did not significantly improve the flexural toughness of the concrete, and neither

wet-dry cycling nor freeze-thaw cycling affected this result. In addition, neither of the two fibre treatments evaluated as part of Phase 2, silane and Duralin, had a significant influence on any of the investigated properties of the flax fibre-reinforced concrete specimens.

Exposure of concrete containing flax fibre to the severe environments studied in this project revealed no evidence that the properties of flax fibre-reinforced concrete will deteriorate over time. This finding, combined with previous work showing that flax fibre is as effective at reducing the occurrence of plastic shrinkage cracking as synthetic fibres, suggests that flax fibre is a technically feasible alternative to synthetic fibres for this application. Before flax fibre will be adopted by the concrete industry, however, it will be necessary to conduct a market study to determine its economic feasibility for this application. Such a study should account for all possible costs, including those associated with modifying farming practices to ensure the production of a consistently high quality flax fibre, extracting the fibre from flax straw, collecting the fibre from farmers, processing, packaging, and marketing. In addition, demonstration projects that incorporate flax fibre in concrete would be helpful to convince the concrete industry of its effectiveness.

The tests described in this report were conducted over periods of exposure that lasted for less than one year. Breakdown of the middle lamella, which binds the elementary fibres into technical fibres, occurred within this time frame, but other degradation mechanisms that occur within the elementary fibre could take place over a longer period of time. This is not expected to cause a deterioration of the properties of concrete containing flax fibre, but it would be helpful to conduct longer-term durability tests on the elementary fibres to confirm this hypothesis and to quantify their degradation in a cement paste environment. Finally, the investigation of additional fibre treatments that would reduce the bond strength between flax fibres and the cement paste would allow the fibres to improve the flexural toughness of the concrete and would thereby open the material to additional applications.

5. Acknowledgements

The significant contributions of Dr. Satya Panigrahi, Department of Agricultural and Bioresource Engineering, and Dr. Ramaswami Sammynaiken, manager of the Saskatchewan Structural Sciences Centre, are acknowledged.

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7. Appendix A: Tabulated Experimental Results for Phase 1

This appendix contains additional tabulated data of the experimental results for Phase 1 of the project.

Table A.1. Lightness value, *L*, for all fibre treatments at all test intervals.

Treatment	Exposure Time (days)					
	0	7	14	28	56	112
Untreated	46.4 (*3.7*)†	46.2 (*4.0*)	45.3 (*8.4*)	48.5 (*8.5*)	51.0 (*3.3*)	57.9 (*5.9*)
Mercerization	46.2 (*5.9*)	49.5 (*4.7*)	47.2 (*8.5*)	55.3 (*4.1*)	57.2 (*3.3*)	60.4 (*4.5*)
Silane	50.9 (*3.3*)	50.5 (*4.4*)	50.8 (*7.2*)	57.3 (*6.1*)	59.5 (*1.4*)	62.4 (*2.9*)
Acrylation	53.8 (*1.7*)	48.2 (*12.3*)	49.8 (*5.6*)	55.7 (*3.6*)	62.4 (*4.3*)	65.8 (*2.0*)
Acetylation	52.0 (*3.2*)	47.1 (*3.1*)	51.6 (*3.4*)	54.2 (*11.6*)	62.5 (*5.0*)	62.0 (*3.1*)

† The italicized values shown in parentheses are the coefficients of variation in percent.

Table A.2. Tensile strength (MPa) for all fibre treatments at all test intervals, including only data for fibres with diameters less than 150 μm.

Treatment	Exposure Time (days)					
	0	7	14	28	56	112
Untreated	134 (64)†‡	141 (57)‡	67 (81)	79 (40)‡	88 (80)	46 (46)
Mercerization	216 (48)	148 (55)‡	60 (42)	94 (-)	51 (85)	29 (-)
Silane	97 (76)	77 (40)‡	89 (41)	80 (45)‡	47 (57)	15 (19)‡
Acrylation	104 (56)‡	110 (53)	116 (54)	41 (50)	93 (76)	29 (7)
Acetylation	134 (62)	125 (73)	58 (25)‡	74 (53)	92 (71)	15 (-)

The number of specimens varied from one to 15, depending on the number of fibres of the desired size available. The average number of specimens was 7.5

† The italicized values shown in parentheses are the coefficients of variation in percent. If no value is shown, only one or two samples were used.

‡ Indicates that an outlier was removed.

Table A.3. Elastic modulus (GPa) for all fibre treatments at all test intervals, including only data for fibres with diameters less than 150 μm.

Treatment	Exposure Time (days)					
	0	7	14	28	56	112
Untreated	8.50 (53)†	11.6 (41)	4.57 (34)	5.98 (25)	5.82 (35)	2.86 (-)‡
Mercerization	12.3 (47)	8.34 (33)	5.62 (69)	4.01 (-)	3.96 (102)	2.73 (-)
Silane	5.40 (70)	5.54 (21)‡	6.27 (29)	4.71 (41)	4.13 (17)	3.76 (113)
Acrylation	6.15 (54)‡	5.72 (23)	5.37 (31)	2.96 (57)	8.79 (68)	1.02 (31)
Acetylation	8.09 (55)‡	5.93 (43)	5.20 (51)	2.86 (21)‡	5.00 (57)‡	1.83 (-)

The number of specimens varied from one to 14, depending on the number of fibres of the desired size available. The average number of specimens was 8.7

† The italicized values shown in parentheses are the coefficients of variation in percent. If no value is shown, only one or two samples were used.

‡ Indicates that an outlier was removed.

8. Personnel and Financial Statement

The experiments for Phase 1 of this project were carried out by two researchers: Mr. Kevin Olubayo, an M.Eng. student, and Ms. Stephanie Walter, an undergraduate student working as a summer research assistant. An M.Eng. report in the Department of Civil and Geological Engineering at the University of Saskatchewan was produced by Mr. Olubayo. Phase 2 was conducted as an M.Sc. project by Mr. Cam Yaremko. Mr. Yaremko's thesis on this topic is in preparation.

A financial statement for this project is being produced by the Financial Services Division of the University of Saskatchewan, pending the receipt of some final project expenses. The statement will be forwarded to the Ministry as soon as it is available.